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ATOMS FOR PEACE

THE INCREDIBLE MARCH of science and technology is terrifyingly demonstrated by the sixteen 500-page volumes shortly to be published covering the recent International Conference at Geneva on the Peaceful Uses of Atomic Energy.

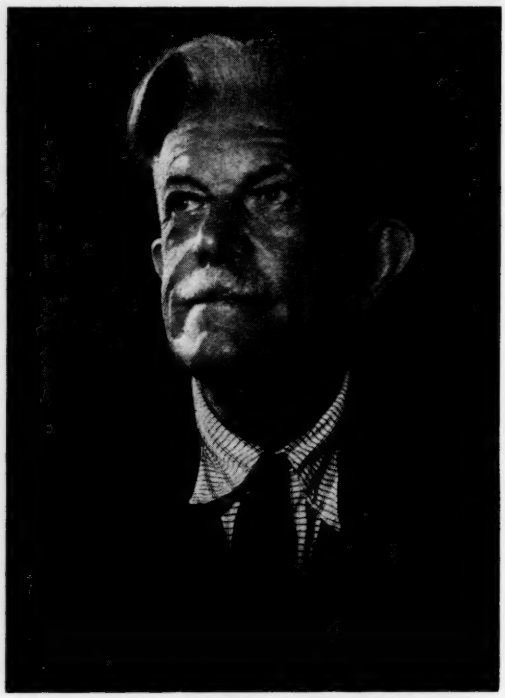
How extensively and swiftly atomic energy developments have progressed since the pioneering nuclear physicists struggled with their troublesome laboratory vacuum system ! As the writer stood before the U.S. Research Reactor at Geneva listening entranced to the attractive young ladies—linguists, who had been trained in a few weeks to tell their story so convincingly as experts—expounding on the mysteries of fission, neutron absorbers, and so on, to the fascinated audience leaning over the blue-glowing water of the Reactor, vivid memories returned of his first reaction on joining the famous Tube Alloys project, as the British Atomic Energy Project was called in the crucial years of the war. The awful secret, to which he had been sworn, the fantasy of the project and the incredible new engineering problems combined to make the early experiences almost nightmarish. As was so often said in those early days of the project ' You do not have to be mad to be a Tube Alloys Worker, but it helps ! ' This was particularly so after frustrating days of leak hunting on what was then a formidably complex system with inadequate pumps and leak detection means. Now all is changed and young scientists quickly become nuclear physicists and take for granted the swift and sure obtaining of 10^{-6} mm. Hg. !

Atomic Energy has profoundly accelerated the development of vacuum techniques and continues to exert, beyond all other applications of high vacuum, the greatest stimulus to its continued development in meeting the demands, for example, of the nuclear accelerator machines, the diffusion plants, isotope preparation, the safety apparatus, and the metallurgy of the fuel and pile materials.

Yet, in spite of the profound technical developments that have taken place in vacuum engineering, it is surprising that the basic elements used by the early vacuum worker remain the same, *i.e.* the McLeod gauge, the oil-sealed rotary pump and the vapour diffusion pump. No startling changes have yet taken place in the fundamental methods of measuring and producing vacuum to parallel those which they have helped to create. This is indeed a great tribute to the early inventors. Yet it is hard to believe, they are so fundamental that the onrush of technology will not displace them, particularly now that vacuum techniques are practised over such a tremendous range of scientific and technical activities.

Signs are not wanting that ideas are becoming possibilities. How soon will those old friends become museum pieces ?

A. S. D. BARRETT.



ADOLF GÜNTHERSCHULZE

Dr.Ing., Dipl.Ing.

Professor Emeritus
of
Electrical Engineering
Technical University, Dresden, Germany

Professor Guentherschulze was born in Hanover (Germany) in 1878. After completion of his general education at a local public school he went to the Technical University, Hanover, as a student of electrical engineering. He was awarded the degree of Diploma Engineer in 1901 and of Doctor of Engineering in 1902. He then joined the staff of the Physikalisch-Technische Reichsanstalt, Berlin, as a research assistant, where he was appointed Professor and ' Member ' of that Institution after 13 year's service. In 1930 he accepted the Chair of Electrical Engineering at the Technical University, Dresden, and remained in this post until his retirement in 1945. In 1949 Professor Guentherschulze resumed his teaching activities when he was called upon to organise a course on semi-conductors at the Technical University, Munich.

Professor Guentherschulze spent part of his career in industry. From 1928-1930 he was the Research Director of Messrs. Osram, Berlin, and later he worked in a similar capacity at Messrs. Telephon-Kable-und Drahtwerke in Nurenberg.

Professor Guentherschulze has contributed a great deal to the progress of science in a variety of spheres. One major study of his was concerned with the rectifying action of oxide films deposited on aluminium and tantalum surfaces which eventually led to a theory of the mechanism of electrolytic condensers. In a similar way he studied the working mechanism of mercury vapour rectifiers and the properties of some semi-conductors such as germanium and silicon. At times the nature of his work carried him into the chemical field to study, for instance, ion exchange phenomena.

One of the most outstanding achievements of Professor Guentherschulze is his contribution to the existing knowledge of cathodic sputtering. This is a product of his systematic investigations into the nature of electrical discharges particularly that of glow discharges. His survey article on cathodic sputtering beginning on the opposite page is another proof of his experience in this subject.

Professor Guentherschulze has constantly endeavoured to give his fellow scientists the benefit of the results of his own work and his total number of publications up-to-date has reached the impressive figure of 180.

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CATHODIC SPUTTERING

an analysis of the physical processes

A. Guentherschulze,† Professor Emeritus, Electrical Engineering Department, Technical University, Dresden, Germany.*

Summary

THE AUTHOR DISCUSSES the results obtained from studying the sputtering effect, first discovered nearly one hundred years ago. He introduces the concept of primary sputtering, subdivided into first and second mechanism, and secondary sputtering. Various factors determining the rate of sputtering, using different metal-gas combinations are investigated. Secondary sputtering, normally observed in a glow discharge and the form of sputtering essentially of technical interest, is defined and the limits of measurement are described. In conclusion, the author remarks on 'chemical' sputtering, on certain engineering aspects and finally on gas adsorption, a phenomenon inherent in the sputtering process.

Sommaire

L'AUTEUR FAIT UNE DISCUSSION des résultats obtenus par l'étude du phénomène de projection cathodique, qui fut découvert pour la premiere fois, il y a une centaine d'années. Il fait apparaître le concept de projection cathodique primaire, subdivisé en premier et second effet, et de projection cathodique secondaire. Les différents facteurs qui déterminent l'importance de la projection cathodique dans le cas de differentes combinaisons metal-gaz sont étudiés dans cet ouvrage. La projection cathodique secondaire, normalement observée dans une décharge ionisante, forme la plus intéressante techniquement parlant, est definie et les limites de mesure sont données. En conclusion l'auteur note qu'un phénomèné inhérent a l'effect de projection cathodique est lié à la projection cathodique chimique ainsi qu' à certains phénomènes mécaniques et à l'absorption des gaz.

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INTRODUCTION

THERE ARE TWO kinds of research scientists. Some will decide on a destination and will never rest until they reach it. Others will take a Sunday stroll and choose an out-of-the-way path for their recreation. If they find it stony and full of obstructions, they will soon turn back, describe it in a rather confused paper and never waste another Sunday on it. The more widely it becomes known that the path is bad, the less it is used and in the end it is deserted completely.

This has been the fate of cathodic sputtering. Nearly one hundred years ago Pluecker discovered that the cathodes in glow discharge tubes lost weight and that the material lost during operation settled down on neighbouring surfaces in the vessel. He called the phenomenon 'Zerstaeubung' (Pulverisation) and this was translated as 'sputtering'.

It is the misfortune of science that every new discovery has to be christened at once and has to be given a name long before one has any notion of its true characteristics. Years later, when the young child of science has grown up, the name does not fit and sounds stupid. Physics has quite a bunch of such misnomers to offer. Take the term gas discharge, for instance: Nothing is 'discharged' in a gas discharge. As long as we call the current passing through a gas-filled lamp 'gas discharge', we should call the current passing through a tungsten lamp 'wire discharge'.

It took 68 years after Pluecker had made his discovery to establish beyond doubt that no sputtering in the sense of a self-initiating process occurs at all.

* MS. received April, 1955.

† Dr. Guentherschulze now lectures at Munich University on the subject of semi-conductors.

Instead, single neutral atoms leave the cathode, freed by the impact of positive ions and a much better name would be ' impact evaporation '. (A practical-minded American might be tempted to condense it to ' Imporation '). However, as an inveterate error is just as venerable as an inveterate truth we will hold on to the old name.

Following the basic discovery another two important facts have been established : Cathodic sputtering in a glow discharge starts at about 300 V and increases linearly with the cathode fall. On the other hand, at very low pressures sputtering, though weak, occurs at potentials almost as low as that of the ionising voltage of the gas.

These findings may serve as the main structure of this paper. First, we will examine the basic process of the emission of neutral atoms by positive ions, called ' primary sputtering ' which is subdivided into a 'first mechanism' at low, and a 'second mechanism' at high voltages. Secondly, we have to consider the fate of the emitted atoms before they are deposited on a surface in the vessel, *i.e.*, ' secondary sputtering ' and finally, in an appendix, we shall have a quick glance at a special offshot—' chemical sputtering ', some engineering aspects and the problem of gas absorption.

PRIMARY SPUTTERING

Equipment for the proper evaluation of primary sputtering must comply with the following conditions:

(1) The ions striking the cathode should have a uniform velocity, *i.e.* they should all be accelerated by the same field. Also the density of the gas present should be low enough for collisions of ions with gas molecules to be neglected. This can be achieved comparatively easily by a gradual reduction of the gas density until the sputtering action is no longer affected by the gas present.

(2) All ions should strike the cathode in the same direction for we dare not hazard the assumption that sputtering is independent of the angle of incidence. As a rule, a perpendicular direction is chosen.

(3) Atoms emitted from the cathode should have no opportunity of returning to the cathode. This qualification calls, (*a*) for the same limitation with regard to the gas density as mentioned under (1), and (*b*) a cathode shape such that no part can ' see ' any other part of it. Otherwise, atoms emitted from one area will settle on another, ' seen ' by the former, and the condition of primary sputtering no longer applies.

(4) Since the incident ions cause the emission of atoms *and* electrons, it is necessary to arrange for the measurement of three different currents: the total current I, the electron current I_- and the ion current I_+, the latter being determined by substracting I_- from I. The sputtering effect, of course, depends only on I_+.

(5) Although most workers state that sputtering is not affected by the temperature of the cathode as long as the latter is not exceptionally high, no harm is done by providing for its measurement.

These are the requirements necessary to obtain infallible data on primary sputtering. It is no use concealing the fact that up-to-date not a single investigation has been reported, where all these requirements have been fulfilled. One reason for this is that, on the face of it, an investigation of secondary sputtering in an ordinary glow discharge is the more attractive proposition for a Sunday stroll.

First Mechanism

Long ago, in 1923, the research laboratories of the General Electric Co., London published a study on ' Cathode Disintegration '. The object was to measure primary sputtering. True to a saying attributed to Roentgen : ' Nature is malicious and elusive and always out to cheat the investigator ', ' Nature ' must have prompted these research workers to employ a wire as cathode in their experiments and, to make matters worse, to use the wire in the form of a closely wound filament. At first sight, a wire looks very tempting. An emission of even the smallest amount of material can be detected simply by recording the change of the electrical resistance of the wire without breaking the vacuum in the vessel.

On closer examination, however, a helix proves to be the worst geometrical form one could choose. Not only is the angle of incidence a different one for each ion, a high proportion of the atoms emitted will settle again on another part of the helix as a matter of course.

On the problem of the first mechanism the statement is made : The intensity of the disintegration of the cathode rises whenever the energy of the positive ions increases and is already appreciable at an energy level not greatly exceeding that required to initiate ionisation of the gas.

That is all that is said on this subject. No specific values are given and no curves are shown.

Another investigation has been published by Fetz. Fetz did not intend to investigate the first mechanism as he was not aware of the concept. His aim was to study the characteristics of primary sputtering as a function of the diameter of a molybdenum wire used as the cathode. A low-density mercury arc served as the ion source. Fetz restricted himself to experiments at one voltage only, *i.e.* at 150 V, which is close to the borderline between the first and the second mechanism. He obtained one value for a flat cathode shown in Fig. 4 for the purpose of comparison.

This is all we know of the first mechanism.

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Second Mechanism

The chief purpose of the G.E.C. investigation mentioned above was a study of the second mechanism. However, the following is stated in that report: Ions are produced everywhere in the vessel and their speed, when they reach the grid (*i.e.*, the sputtering cathode) depends on the difference between the potential in the region where they are formed and that of the grid.

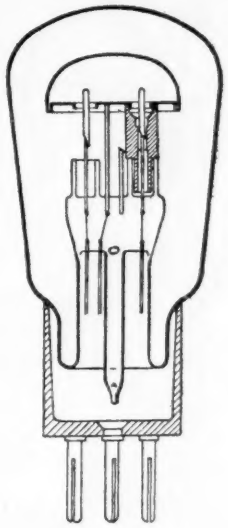
This makes it clear that so many of the conditions, laid down on page 361, have not been fulfilled that the individual values obtained have no general application.

However, the following conclusions are safe :

(1) The rate of disintegration is independent of the temperature of the cathode.

(2) The rate of disintegration varies according to the nature of the gas.

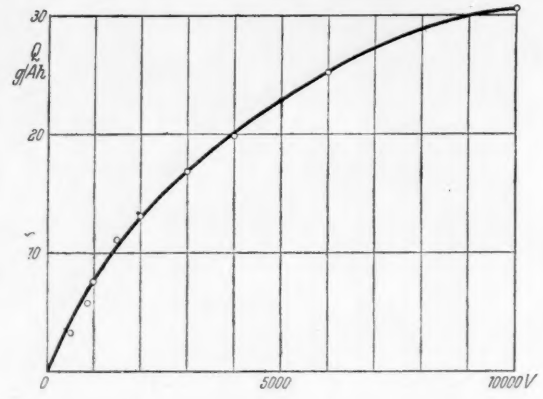
The gases investigated, given in the order of a rising rate of disintegration, are : hydrogen, helium, nitrogen, neon, mercury vapour and argon. So far as helium is concerned there must have been a mistake as will be seen below. Of course, these observations are only valid for tungsten, the sole cathode material used.



Z. Phys., 1930

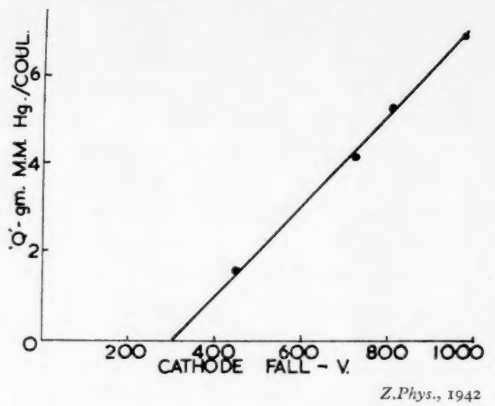
Fig. 1. Sectional drawing of a Raytheon glow discharge helium rectifier operated with a.c. The cup-shaped electrode is faced by two tantalum rods forming the other electrode.

Two investigations have been published by A. Guentherschulze and K. Meyer. In the first, copper and silver cathodes were employed in hydrogen, nitrogen, neon and argon atmospheres. In the second, cathodes of 16 different metals were operated in mercury vapour. The ions created in a 3-Ampere discharge produced by an oxide-coated glow cathode were accelerated in a Langmuir dark space, involving a negligible number of collisions, towards the cathode which was a flat disc suspended from a spring balance. All requirements were complied with in these experiments, except one : Only the total current was



Z. Phys., 1931

Fig. 2. A typical record of primary sputtering showing the measured values of the quantities of tantalum sputtered as a function of the voltage.



Z.Phys., 1942

Fig. 3. A typical record of secondary sputtering showing the measured values of the quantities of copper sputtered in a hydrogen discharge.

measured.

Helium showed an entirely unexpected behaviour. Up to the highest voltage employed in this investigation, *i.e.*, up to 800 V, no trace whatsoever could be found of the sputtering of silver, a metal which in all other gases sputters generously. There is another instance of this in the engineering field. At that time Raytheon rectifiers came on the market, see Fig. 1. The rectifiers worked on the glow discharge principle. One of the electrodes was shaped in the form of a cup and the other consisted of two tantalum rods. The apparatus was filled with helium and operated with alternating current at 500 V. After a service life of more than 1,000 hours the surface of the rods did not show any change which could be attributed to sputtering, but when the helium had been replaced by neon, the rods were completely destroyed after a short time. Thus we can assume that no sputtering whatsoever takes place in helium.

In spite of this, helium is absorbed in a helium discharge. The helium seems to penetrate into the cathode without causing sputtering. Perhaps I should emphasize that the absence of sputtering can only be proved if the helium is absolutely clean. As the ionisation voltage of almost all other gases is much lower than that of helium a very small concentration of impurity molecules in helium leads to a comparatively large number of impurity ions. Thus if anybody should claim to have discovered sputtering in helium it would be advisable to check on the purity of the gas used.

There is a strange sequence to the facts reported in these two investigations. When I was asked to write this review I looked up the two 25-year old papers and noticed two things. First, there is a discrepancy between Fig. 2, recording the sputtering of tantalum against voltage up to 10,000 V and presenting a typical picture of primary sputtering, and Fig. 3 which shows a typical curve of secondary sputtering of copper in a glow discharge. Fig. 2 starts at 0 and Fig. 3 starts at 305 V. Second, the curve of Fig. 2 looks like a square root function with a slight irregularity near 0. I decided to plot the measured values of Fig. 2 against the square root of the voltage, *i.e.* against the velocity of the ions, or, in an alternative interpretation, against their momentum in arbitrary units. Fig. 4 shows the result. With an accuracy, seldom reached in the measurement of sputtering all points lie on a straight line which starts at 13.3 km./sec., corresponding to a voltage V_0 of 190 in the particular case. I then hastened to plot all other

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combinations of our two investigations accordingly and without exception I obtained the same conditions in each case. All combinations obey the law

$$Q = A\,(C\text{-}C_0) \qquad (1)$$

where Q is the sputtered material in g./coulomb (total current), A is a constant depending on the metal-gas combination used and C and C_0 are the velocities of the striking ions in km./sec.

So far it was generally agreed that the sputtering rate depends on the energy of the ions. Now we are suddenly confronted with the fact, that it depends on their momentum. However, there seems to be a flaw in this new form of presentation : Q is determined using the total current. The electron current is not taken into account. As the latter increases with the voltage we have still to look for conclusive evidence. Fortunately, at a much later date in fact, I have also measured the emission of electrons produced by ions incident at energies up to 3,000 volt. And indeed plotting these values *versus* the momentum of the ions yields again straight lines above a certain threshold value. Admittedly, I conducted only one investigation with the aim of establishing both the sputtering rate and the electron emission rate, using the combination of copper-argon. No doubt, I must have been on a Sunday stroll myself at the time. But, it so happens in this case that the threshold values are identical.

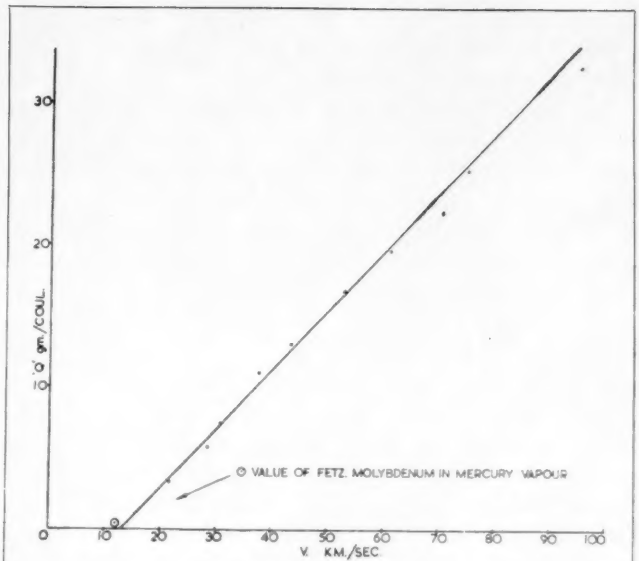


Fig. 4. Quantities sputtered shown as a function of ion momentum by plotting the measured values of Fig. 2 against the square root of the voltage.

Thus we have the situation illustrated in Fig. 5. I is the total current measured. By subtracting from I the electron current which starts at the threshold B and increases linearly, one obtains the ion current I_+. If now the measured quantity of material sputtered, Q, is multiplied with the factor I/I_+, a new curve can be plotted for $Q_{\text{corrected}}$ which has a slight bend in the upward direction.

These considerations demonstrate how much still remains to be done, and bring home the point that it is absolutely necessary to measure sputtering *and* electron emission in these investigations. Electron emission incidentally, is very sensitive to variations in the conditions of the surface and it is no use to rely on values which were obtained by another observer in an apparatus of different design. Also, electron emission, too, seems to have two mechanisms. One,

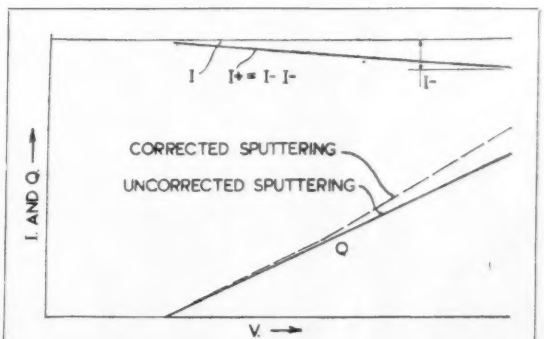


Fig. 5. Graph showing the method of correcting the values of the amounts sputtered obtained by Equ. 1 taking into account the emission of the electrons produced at the voltages applied.

chiefly occurring at low voltages, consists of photo-emission, the other, occurring at higher voltages, consists of momentum emission.

K. Meyer's investigation in a mercury vapour atmosphere produced another rather surprising result : If the amount sputtered is expressed in terms of the number of atoms N emitted by one ion striking the surface (total current), the eight metals, aluminium, silicon, manganese, iron, zirconium, molybdenum, tantalum and tungsten, all follow the same equation :

$$N = 62.9 \times 10^{-3} (C-13.3) \qquad (2)$$

where C has the dimension of km./sec. The remaining eight metals listed in Table I showed a higher amount sputtered, the easily amalgamating metals such as silver and copper yielding the highest values. The measurements taken on the metals listed in Table I were effected at one voltage only, *i.e.* at 1,000 volt. At this voltage Equ. 2 gives a value of 1.08 atoms/ion for the amount sputtered. As can be seen all metals of Table I show a higher value. The amount sputtered grows as the tendency towards amalgamation increases. Obviously, amalgam formation favours sputtering.

Table II contains the constants of Equ. 1 for gases other than mercury vapour.

Some time later Penning and Moubis investigated primary sputtering in a glow discharge. The cathode was a water-cooled copper tube situated between two magnet poles. The magnetic field reduced the length of the dark space to such an extent that the collisions of the ions with the gas molecules could be neglected.

Experimenting with copper in argon they found a value of 1.7 atoms emitted per ion (total current) at 500 V. Using the constants of Table II the value is 1.58 atoms/ion* at 500 volt.

SECONDARY SPUTTERING

Whereas in primary sputtering the gas density must be kept sufficiently low to be neglected, it is a predominant factor in secondary sputtering, normally observed in glow discharges. All work on glow discharges has been haunted by an evil spirit, called gas pressure. All specialists know that gas discharges in general and glow discharges in particular do not depend on gas pressure; they depend on gas density. But as direct measurement of gas density is impracticable and measurement of gas pressure is easy, they substitute pressure for density soothing their nagging conscience with the assertion that the temperature is ' almost ' the same throughout the vessel and only ' very little ' above room temperature. Unfortunately, however, the discharge heats the gas, the anode and, especially, the cathode. If the electrodes are placed inside the vessel they give up their heat to the gas. Temperatures of 300°C in one region of the gas

Table I

Number of Atoms Sputtered by a Striking Mercury Ion (Total Current) at 1,000 V.

Metal of Cathode	*Number of Atoms Sputtered N*	*Metal of Cathode*	*Number of Atoms Sputtered N*
Nickel	1.10	Platinum	2.17
Beryllium	1.28	Palladium	2.62
Cobalt	1.57	Copper	2.90
Chromium	1.80	Silver	5.10

Table II

Constants of Equation 1 for Silver and Copper in Different Gases.

Gas	C_o Km./sec.	V_o Volt	A Atoms/Ion
Silver Cathode			
Argon	21.8	101	110×10^{-3}
Neon	13.3	19	23.7×10^{-3}
Nitrogen	28.5	121	62.8×10^{-3}
Hydrogen	70	52	39.2×10^{-3}
Copper Cathode			
Argon	13.3	38	45.2×10^{-3}

* Very interesting research work on primary sputtering is presently being done by Gottfried K. Whener in Ohio. Some of it has been published, but I should like to postpone its review until it has been finished.

atmosphere and of room temperature in other regions are no rare occurrence and cause errors in measurement of up to 100%. It is no wonder that so many investigations failed to establish simple laws. There is only one way out of this difficulty. The electrodes should be made part of the walls of the vessel and be kept at room temperature by energetic cooling. Further, the current density should be kept so low that the temperature rise of the gas could be either neglected or, failing that, determined accurately.

Turning now to the mechanism of glow discharge sputtering, we are faced with a horrifying maze of most complicated conditions. In a normal cathode fall the ions suffer some 50 collisions while they are accelerated in the dark space. Thus they strike the cathode with an energy which can be of any value between nil and almost the maximum energy corresponding to the cathode fall. If we look at this in the light of the results obtained for primary sputtering, where it was established that sputtering starts at a certain threshold and depends for its intensity on the momentum of the ions, we realise some of the difficulties we are faced with. Others stem from the fact that the energy of the striking ions is by no means proportional to the cathode fall in the case of abnormal discharges. As the cathode fall increases at any given gas density, the length of the dark space decreases to one fifth of its length under normal conditions. Therefore the number of collisions diminishes but the striking velocity rises steeply, out of proportion to the rise of the cathode fall.

Examining the behaviour of the emitted atoms we have to distinguish between the following conditions:

(1) The energy of the atoms leaving the cathode is of the order of their energy at room temperature.

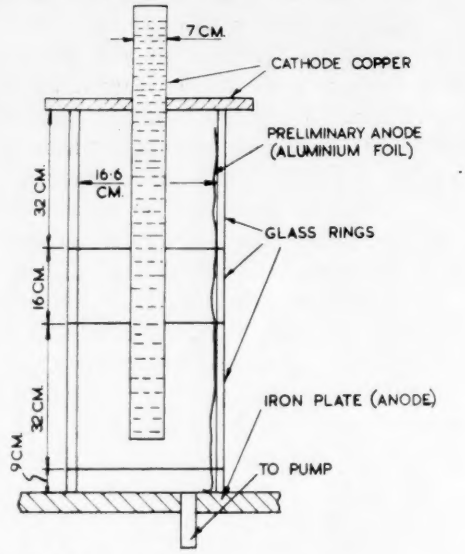
(2) The energy of the atoms leaving the cathode is appreciably higher. This applies when the striking ions have high energies.

(3) The mass of the atoms is of the same order as that of the gas molecules. In the case of the copper-argon combination, for instance, copper equals 63.57 and argon equals 40.0.

(4) The mass of the atoms is appreciably greater than that of the gas molecules. In the case of the platinum-hydrogen combination, platinum equals 195.7 and hydrogen equals 2.016.

The two examples quoted represent the two extremes : Copper atomes of low energy in argon and platinum atomes of high energy in hydrogen. With regard to the former it has been shown that as a rule the emitted atom loses its initial direction at a distance d from the cathode equal 2.5 x the mean free path. It then behaves like a normal diffusing particle. We will call this region the ' neutral zone ' of diffusion. From this zone the atoms proceed to the various surfaces of the vessel in numbers which are inversely proportional to the distance of the respective surfaces, *i.e.* nearly all return to the cathode. The flow lines formed by the diffusing particles have the same relative density and direction as the lines of an electric field, we would obtain if, for instance, we were to arrange for the neutral zone to be charged positively and all chamber and cathode surfaces to be charged negatively. We are now confronted with the same sort of problem as that presented in the calculation of the capacity of an electrostatic condenser. Three geometries offer themselves : (1) Concentric spheres, (2) coaxial cylinders and (3) parallel plates. In the case of an electrode arrangement according to (2) and (3), the distance between the electrodes should be very small compared with all other dimensions as it is evidently the simplest method to make the anode the collecting surface. For the glow discharge to be undisturbed the smallest distance is : dark space plus half the depth of the negative glow. In the platinum-hydrogen combination, for instance, the normal dark space is 1.0 cm. at a pressure of 1 mm. Hg. As the dark space is inversely proportional to the gas pressure it becomes 10 cm. at 0.01 mm. Hg and the minimum distance in question, 15 cm. Thus, we would need tubes of nearly 50 cm. diameter and 150 cm. length or alternatively circular plates of 150 cm. diameter, kept 15 cm. apart by means of a glass ring of identical diameter as the electrodes,—hardly a practical proposition.

If we wish to measure secondary sputtering up to a level of several thousand volt, we have to employ very low gas densities, because at a given density the current increases almost as the fourth power of the



Z. Phys., 1942

Fig. 6. First laboratory set-up designed for basic experiments on secondary sputtering. The tubular cathode made of copper was coated with the metals to be investigated.

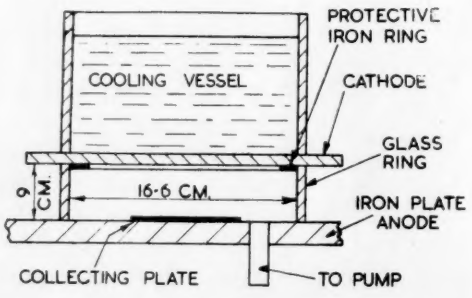


Fig. 7. An improved design of the vessel shown in Fig. 6. The disc-shaped cathode facilitated easy application of the layers of the metals to be investigated.

cathode fall and at constant cathode fall decreases as the second power of the gas density. Therefore, if the gas pressure is not kept very low, the gas heats up to such a degree that our act of deceit, so persistently worshipped, of substituting pressure for density is exposed and can no longer be tolerated. Thus we come up against the deplorable fact that all means of measuring secondary sputtering without any flaws and loopholes are stopped just above a thousand volt due to technical difficulties.

Up to the last war not one of the many investigators took due notice of the necessity for these precautions. Strong heating of the gas and intricately shaped surfaces, collecting the atoms by design or accident, were a common feature in the experiments. The position is this : If we have a parallel plate at a distance D from the cathode and the neutral diffusing zone at a distance d from the cathode, then $d/(D+d)$ is the fraction of the atoms which is collected by the anode and since $d = 2.5$ x the mean free path is proportional to the gas density, $d/D+d$ is inversely proportional to the gas density. If we further assume that the gas in the dark space is heated to 300°C, that the vessel is large and the cathode comparatively small, then the gas density in the dark space can be reduced to half its original value without an appreciable increase of the pressure and in consequence the measured value of the sputtering will be 100% too high. That is why not a single absolute value of secondary sputtering obtained by properly defined methods of measurement became available before the war.

During the war, although I had no assistants to help me, I attempted a little more than just a Sunday exercise. I started my experiments using the vessel shown in Fig. 6. The cathode was a copper tube of 70 mm. diameter, closed at the bottom by a copper disc. Its upper part was soldered to a copper plate of 200 mm. diameter. The tube was filled with water which was kept at room temperature. The copper plate served as the lid for the discharge vessel. The latter consisted of 4 glass rings of 166 mm. diameter and of a height as shown in Fig. 6. The joints were sealed vacuum-tight with wax. The whole assembly rested on a thick iron plate which also formed the

anode. In order to obtain a homogeneous current density along the cathode a small strip of aluminium foil was connected to the anode base and placed along the wall as shown. When the glass rings became conducting due to the copper depositing on them, contact was made with the aluminium strip and the whole surface of the glass rings became the anode. The experiments were continued until the sputtered layer was opaque. This took from 5 to 23 hours. Subsequently the chamber was dismantled, the copper deposited on the cylinder in the centre was dissolved with nitric acid and an analysis made. After a short time this arrangement was improved. The inside of the glass rings were entirely covered with bright aluminium foil and all the copper was deposited on it. After a run a ring of the foil in the centre of the column was cut away and the copper deposited on it was weighed without difficulty after it had been removed mechanically from the foil with the help of caustic soda solution.

In the above arrangement it was rather difficult to coat the cathode with the various metals it was intended to investigate. For that reason new experimental equipment was developed, see Fig. 7, and the old apparatus was used only for the calibration of the new set. Here, the cathode consists of a thick circular disc of the metal examined and is cooled by a large container filled with water. The cathode rests on a glass cylinder of 16.6 cm. diameter and 4.0 cm. height. It proved necessary to provide for a protective ring, 1.0 cm. in width, placed at 1 mm. distance from the cathode. Without it, the metal sputtered onto the glass walls would have soon made contact with the cathode, would have become part of it and consequently the glass rings would have been destroyed by excessive heating. This assembly rested on a thick iron plate serving as the anode. In the centre of the anode a thin circular copper disc of 8.0 cm. diameter collected the sputtered atoms. Originally it was assumed that the diffusion flow lines would be parallel at least near the axis of the vessel and thus sputtering in that area would be free from any disturbing influences. But, looking closer at the results obtained with the arrangement of Fig. 7, it was discovered that the amount deposited on the copper

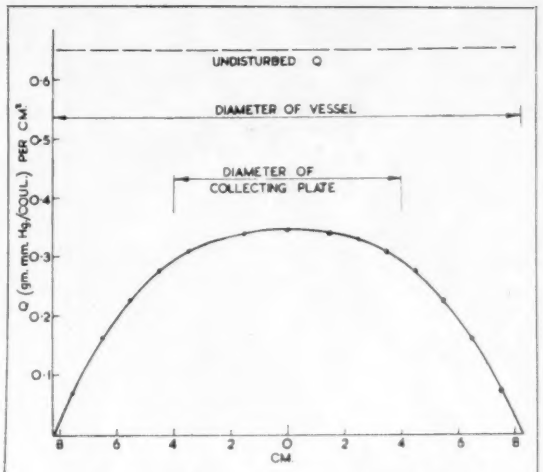


Fig. 8. The graph shows the distribution of the sputtered deposits on the anode surface obtained in an experiment sputtering copper in a hydrogen glow discharge at 875V.

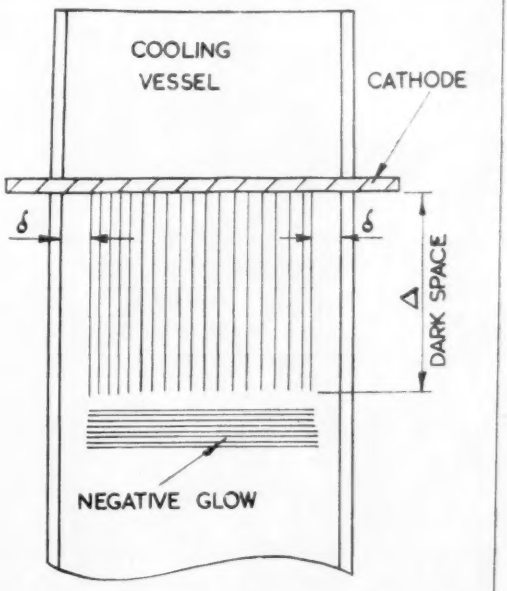


Fig. 9. Effect of operation at higher voltages. The discharge is repelled from the chamber walls and a dead zone (δ) develops. △ is the dark space.

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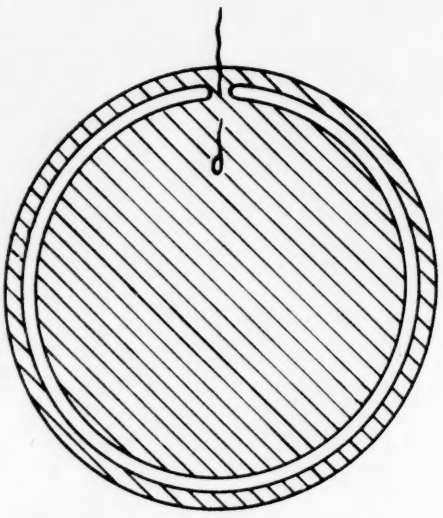

Fig. 10. Example of the effect of the dead zone. An iron disc showing a circular slot after exposure to secondary sputtering at a high cathode fall for two days. (1,000 V.)

disc was much too small to be correct. In fact, there was a discrepancy of 76.7% and a correction factor of 1.767 had to be applied to arrive at the true value. To ascertain the distribution of the deposit across the whole collecting surface, aluminium foil was placed on the anode. When sufficient copper had been deposited, the foil was cut into small rings removed from the anode and the copper of each ring determined separately. Fig. 8 shows the values obtained in experiments at 875 volt.

At higher voltages a further disturbing effect makes itself felt : As Fig. 9 demonstrates, the discharge is repelled from the walls of the chamber due to electrostatic forces and a dead zone develops on the periphery of the cathode. In the extreme case, at relatively high voltages, we are up against the well-known phenomenon of cathode rays which originate from a point almost on the axis of the discharge tube, leaving the rest of the cathode surface dead. This can be fatal for cathodic sputtering. The dead zone is much nearer to the neutral diffusion zone than any other surface. The larger it grows compared with the live part of the cathode, the more atoms it absorbs. The inevitable conclusion is that the quantity of atoms collected on the anode is subject to the voltage applied. It reaches a maximum at a certain level and decreases towards zero at higher voltages. A. v. Hippel observed the same behaviour but he did not know the true cause of it and gave the following explanation : At higher voltages, he said, the ions penetrate so deeply into the cathode that sputtering of the cathode surface is greatly reduced. He does not disclose how the ions thrust by such enormous forces into the interior of the cathode manage to avoid blowing up the cathode in the process. This effect is only slightly minimised, if the arrangement is changed and the cathode is suspended in the centre of the discharge chamber in a position far away from the walls. The following experiment illustrates that neatly : A circular iron disc, 0.1 mm. thick, was suspended in the middle of a large glass bulb and exposed to a sputtering glow discharge of 1,000 volt. After two days it had the appearance shown in Fig. 10. Due to the sputtering effect the iron in the area 3 mm. inside the edge had been eaten away completely. Since the edge of the disc was dead and consequently all atoms ejected near it had been drawn off to that area, a circular slot was produced which was 2 mm. wide. This ' marginal effect ' is another phenomenon which makes it very difficult to operate at higher voltages.

With the equipment shown in Fig. 6, I studied the copper-hydrogen combination and obtained the only set of quantitative values in existence for any combination. It reads

$$Q = 1.028 \times 10\,(V\text{-}303)\ \frac{\text{g. mm. Hg.}}{\text{Coul. cm.}^2} \qquad (3)$$

During the final stages of the war I studied many more combinations but when I began to compile the data, my laboratory at Dresden was destroyed and all my notes with it.

COMPARISON OF PRIMARY AND SECONDARY SPUTTERING

Fig. 4 shows that primary sputtering is a linear

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function of ion momentum. Fig. 3 shows that secondary sputtering is a linear function of the cathode fall. At a first glance, these appear to be contradictory statements and this would be so if the energy of the striking ions was proportional to the cathode fall and if the distance of the neutral zone was independent of the voltage. However, as I have emphasised before, the energy of the ions rises a good deal more steeply than the cathode fall increases because the length of the dark space diminishes in the process and the neutral zone moves away from the cathode as a result of the higher energy of the emitted atoms. Both effects together would cause an upward bend in the curve of Fig. 3 if sputtering were a linear function of the energy of the striking ions. This upward bend seems to compensate for the downward bend of the square root function of primary sputtering plotted against the energy of the ions.

The fact that secondary sputtering represents a diffusion phase added to the state of primary sputtering, appears to offer a new means of measuring primary sputtering,—such as the following : We measure undisturbed secondary sputtering between two parallel plates, a distance D apart in a gas atmosphere of 1 mm. Hg, determine the distance d=2.5 x the mean free path from the cathode, multiply the observed amount of sputtering by D/d and thus obtain the amount of primary sputtering. Again we are hampered by the lack of available data. The only reliable values of secondary sputtering we possess apply to copper in hydrogen. This combination has never been used in experiments on primary sputtering nor does it fulfil the condition mentioned on page 366 (No. 3) that the metal atoms and the gas molecules are of equal mass. The combination, nearest to it in character, which was investigated for primary sputtering, is copper-argon. All authors agree that the amount of secondary sputtering in argon is at least double that in hydrogen. Therefore, we can take the value for copper in hydrogen, determined in the apparatus of Fig. 6 at 1,000 V with a distance of 48 mm. between the electrodes, and double it. This gives us a value of 1.44 g./coul. at a pressure of 1 mm. Hg. At this pressure and 20°C temperature the mean free path of an argon atom in argon is 0.0518 mm. Thus d equals 0.13 mm if we assume that the value is approximately the same for a copper atom in argon and the amount of primary sputtering is determined by the product 1.44 x 48/0.13=550 g./coul. The amount collected on the anode was determined experimentally and found to be 7 g./coul. That is a most surprising result but it holds good generally. The figure of 550 g./coulomb appears to indicate that on the whole the calculated values for secondary sputtering—inaccurate though they may be—are very nearly of the same order as those for primary sputtering whereas according to the law of diffusion they ought to be at least two orders of magnitude smaller.

It may be useful in this connection to employ an analogy comparing the sputtering of a cathode in the primary process with the bombardment of a 'rock' and the sputtering of a cathode in the secondary process with the bombardment of 'fine sand'. As defined before, primary sputtering shows no diffusion phenomena because the density of the gas atmosphere present is too low, but in secondary sputtering diffusion takes place and the 'fine sand' actually constitutes evaporated atoms diffusing back to, and settling down on, the cathode, intermingled with diffusing gas molecules and only slowly assuming the form of a solid metallic layer with a normal lattice structure. This interpretation appears justified as it is corroborated by two observations : (*a*) Several authors point out that they found the cathode covered with a dustlike layer which could be removed easily.

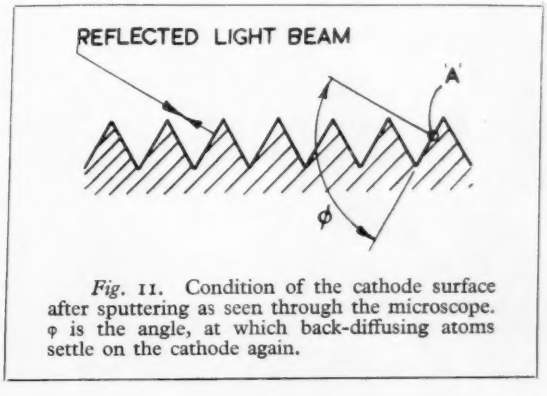

Fig. 11. Condition of the cathode surface after sputtering as seen through the microscope. φ is the angle, at which back-diffusing atoms settle on the cathode again.

(*b*) All authors agree that there is a time lag between switching on the current and the actual starting of sputtering. No doubt, in some cases, this time lag could be caused by the presence of an oxide layer which has to be removed first and sputters slowly. But the effect is the same even if there is no trace of an oxide layer. Thus, one may deduct that this period constitutes the time necessary to transform the ' rock ' into ' sand ' and can assume that the value of 550 g./coul. represents the amount of sputtered

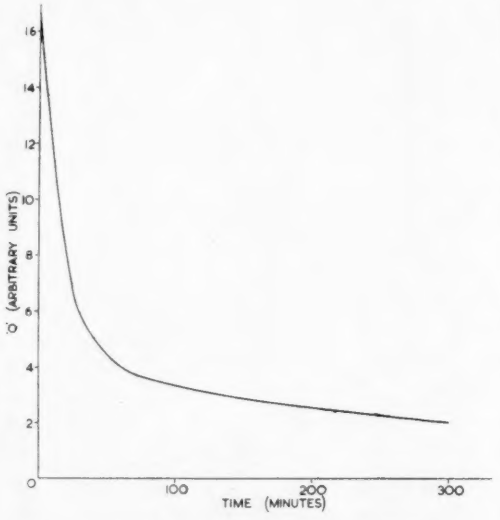

Fig. 12. The cathode surface condition (see Fig. 11) causes a reduction of the amount sputtered with time. The graph refers to lead sputtered in hydrogen at 1,400 V and 0.23 mA/cm².

atoms produced by the primary process and supplied to the neutral diffusion zone, from which they diffuse to the various surfaces in numbers inversely proportional to their distances. According to this conception the energy available for each sputtered atom would be 4.3 eV in the particular case. Considering that the heat of evaporation is 3.0 eV for copper a sputtering mechanism of this kind would have an efficiency of 70%, a very high figure.

At any rate, primary sputtering at a very low gas pressure and primary sputtering in a glow discharge with large back diffusion are two entirely different processes. That presents another rather troublesome complication.

Surface Condition of the Cathode and Striking Angle

Primary sputtering roughens the smooth surface of the cathode just a little. Secondary sputtering produces a completely different effect. Here, the metal atoms diffusing back form microscopically small cones on the cathode. Fig. 11 gives a diagrammatic cross section of these deposits. If you look at the deposit in a direction normal to the surface it appears dark. But, if the light source is orientated as shown in Fig. 11, and the surface viewed in the direction indicated, then it appears very bright. The angle of maximum reflection has been measured by Guentherschulze and Tollmien. Its value differs for each metal and depends, in addition, on the orientation of the metal crystals in the surface.

This phenomenon associated with back diffusion interferes seriously with the measurement of primary sputtering. Suppose an ion strikes the cathode surface at point A, then the atoms which leave point A within the angle φ are stopped before they can escape. Fig. 12 shows the large decrease of the sputtering rate of lead with time owing to this effect. Thus we are caught in a trap, from which there is no way out. In the circumstances, we should measure sputtering immediately after starting the experiment, *i.e.*, before the cones have formed, but we cannot do so because of the time lag mentioned before. Fortunately this phenomenon is completely absent in the case of gold, iron, nickel and platinum and little effective in the case of silver and copper. At any rate further examination reveals that it would not be much use to have the cathode surface especially highly polished before the start of the experiment. A surface is considered highly polished if the polishing scratches are appreciably smaller than the wavelength of visible light, say, 5,000 Å. But the size of an atom is only of the order of 1 Å. A highly polished surface looks to an atom therefore like a rocky hillside and that is why the ions strike the surface at all angles.

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Velocity of the Emitted Atoms

Nothing is known about the velocity at which ' rock ' emission takes place in the course of primary sputtering and its measurement appears difficult. But secondary sputtering can be made to record the emission velocity automatically on the walls of the chamber. Using a cylindrical glass chamber as depicted in Fig. 9—covering the walls with aluminium foil—it can be shown that, in the case of normal diffusion, the deposit on the walls decreases logarithmically with the distance from the cathode. Fig. 13 gives such a graph. But if heavy atoms are shot at great speed into a light gas (lead into hydrogen, for instance) then the deposition process is governed by a quite different law.

Summing up the results of the various experiments it can be stated.

In hydrogen and at voltages up to 1,000 or possibly even up to 2,000, the atoms of most of the investigated metals can be assumed to proceed in the chamber by way of normal diffusion as long as allowance is made for the fact that the neutral zone withdraws from the cathode as the voltage increases. Above 2,000 volt quantitative measurements are no longer possible on account of the difficulties mentioned before.

NOTES ON SPECIAL ASPECTS

Chemical Sputtering

The gas discharge represents a very effective means of building up chemical compounds. In a glow discharge in oxygen with a platinum cathode, platinum atoms are liberated, oxidised by active oxygen ions and deposited on the walls as platinum oxide (PtO_2).

Compounds may be formed in this manner which cannot be obtained by normal chemical procedures. The behaviour of arsenic, antimony, bismuth and tellurium in hydrogen was investigated fairly closely. All these materials form gaseous hydrides such as arsenic tri-hydride, antimony tri-hydride, etc. These molecules diffuse throughout the chamber without being deposited anywhere. When they collide with an ion they disintegrate and the respective metal atom proceeds preferably to the nearest wall. Here it may be transformed again into the hydride, should the wall be hit by positive ions in that particular place. The pattern of such a deposit is entirely different from that produced by normal sputtering and can be immediately recognised as ' chemical ' at first sight.

Engineering Problems

The engineer looks at sputtering with mixed feelings. On one hand he welcomes it as a means of covering surfaces with very thin layers of metals, on the other he dreads it as a possible source of trouble,

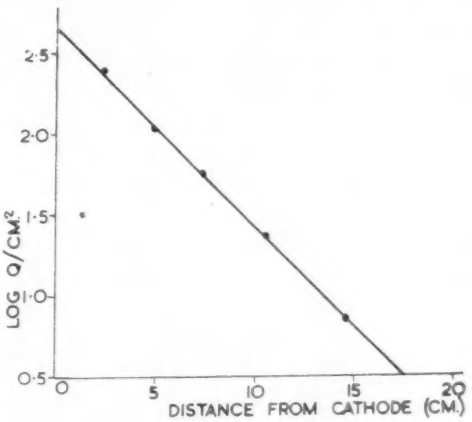

Fig. 13. Deposition on the walls decreases logarithmically with the distance from the cathode. The data refer to the secondary sputtering of copper in argon at 1,000 V and 67 mA for 43 hrs. Total amount sputtered—982 mg.

especially as it destroys oxide-coated glow cathodes.

As to the former, sputtering competes with thermal evaporation in vacuum. The evaporation method has the advantage that the deposited layers are free from gas. Coating by sputtering is preferable only where the temperature required to obtain the necessary vapour pressure is impracticably high.

The rate of sputtering is highest in argon. If the danger of oxidation is present, hydrogen is the best gas to use. The metals most suitable for deposition

by sputtering are : Gold, silver and copper. The electrical resistivity of very thin layers is quite different from that of the bulk metal.

There are cases where all sputtering effects must be suppressed—one reason, for instance, may be that the resulting deposit would short-circuit the electrodes. There are two ways to achieve this. One is to use aluminium or magnesium as electrode materials. Both are reputed to sputter very little, but it is not quite correct to say this. Both materials sputter amply as long as their top surface really consists of aluminium or magnesium. The rate of sputtering is low only, if an oxide film of the metal constitutes the actual sputtering surface. In atmospheric air both metals are always covered with oxide layers. They may be operated for days under a heavy load in clean argon or neon without any sign of sputtering until suddenly, in a matter of minutes, the walls of the discharge vessel become opaque as a result of the deposition of a bright metallic layer. This can be avoided by employing a rare gas containing a small amount of impurities such as oxygen or water vapour which may serve to renew the slowly sputtered oxide layer. Deposits obtained from oxide sputtering do not affect operation, since they act as insulators. An alternative possibility is the use of helium. In this case however the gas must not contain the slightest impurity, not even an admixture of another rare gas and in some instances it is impossible to guarantee extreme purity.

Gas Absorption

It is a known fact that all metals exposed to a gas atmosphere slowly absorb gas molecules. It is evident therefore that the sputtered deposits contain gas and that the amount absorbed during the process increases with the gas density prevailing in the vessel. At the same time small gas atoms may be shot into the cathode at higher voltages. In general it is rather difficult to isolate the two effects. Helium is the only exception. A silver cathode operated in helium at 800 V absorbed 0.053 mg. of the gas in 345 mA minutes without showing any signs of sputtering.

However, the effects of this latter type of bombardment can never become cumulative as the ' invaded ' layer is continuously removed by the sputtering process. Gas molecules taken up by the electrode surface from the gas atmosphere can be removed by heating in vacuum but gas molecules which penetrate into the body of the metal by this form of bombardment cannot be removed in this way, or at best, only partly. In the former case the gas molecules move along the grain boundaries—a process which is reversible. In the latter case they are jammed so tightly into the lattice that no diffusion is possible.

There are only a few quantitative values available for the amount of gas absorbed by molecules during condensation. For instance, we know that 144 silver atoms absorbed one neon atom in the process. This is indicative of the order of magnitude of the absorption of other gases as well, except of mercury vapour which has not yet been investigated.

CONCLUSIONS

This survey will have revealed to the reader the deplorable state of our knowledge on cathodic sputtering. Admittedly one of its aims is to act like an ion striking the physicist in order to sputter enough experiments to clear up the whole problem. It seems such a shame to me that the science of physics with all its wonderful achievements has not succeeded yet in overcoming the difficulties presented by this phenomenon, interesting as it is. I know I ought to have attempted it myself but since I left Dresden, 10 years ago, I have had no opportunity for doing any practical research.

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the properties of some

REACTIVELY SPUTTERED METAL OXIDE FILMS

L. Holland &
G. Siddall.

*Research Laboratories, Edwards High Vacuum Ltd., Crawley, Sussex**

Summary

SOME OF THE RECENT applications of reactively sputtered metal oxide films for electrical and optical purposes are described. A series of conducting oxides approximating to the compositions cadmium oxide (CdO), tin oxide (SnO_2), indium oxide (In_2O_3) and iron oxide (Fe_3O_4) have been prepared and their respective properties are discussed in relation to their use as transparent conducting coatings on glass windows. It is shown that the electrical conductivity of sputtered cadmium oxide films decreases as the degree of oxidation of the deposit rises. The conductivity of a highly oxidised deposit can be greatly increased by heating the film in air to cause a partial dissociation of the oxide. Cadmium oxide films of the highest conductivity may also be produced directly by careful control of the glow discharge and residual gas mixture during sputtering. The method of preparing gold films of compact structure by using bismuth oxide substrates due to Preston and Gillham[2] has been investigated, and their results confirmed. Gold films of 100 Å thickness on bismuth oxide are shown to have a low resistivity (10 ohm per square) and a high optical transmission (75 per cent.). Values of the resistivity and temperature coefficient of resistance of gold-films on bismuth oxide are given for films 20/400 Å thick in the temperature range 15/150°C. It is shown that the temperature coefficient decreases with the film thickness and can be related to a rise in the resistivity of the deposit. The optical transmission and resistivity of typical coatings of each of the above materials are given in tabular form.

Sommaire

UNE DESCRIPTION est faite des applications récentes à l'électricité et l'optique de la formation de films d'oxydes métalliques déposés par projection cathodique réactive. On a formé une serie d'oxydes conducteurs, CdO, SnO_2, In_2O_3 et F_3O_4. Une étude est faite sur leurs propriétés respectives en tant que revêtements conducteurs et transparents pour vitres. On montre que la conductivité des films d'oxyde de cadmium déposés par projection cathodique décroit lorsque le degré d'oxydation du dépôt augmente. La conductivité d'un dépôt fortement oxydé peut être augmentée considérablement par chauffage du film dans l'air pour donner lieu à une dissociation partielle de l'oxyde. Des films d'oxyde de cadmium de très grande conductivité peuvent aussi être formés directement par un contrôle sévère de la décharge ionisante et du mélange de gaz résiduel pendant la projection cathodique. La méthode consistant à préparer des films d'or de structure compacte en se servant d'un support d'oxyde de bismuth, méthode due à Preston et Gillham, a été soumise à investigation et ses résultats confirmes. On montre que des films d'or de 100 Å déposés sur de l'oxyde de bismuth ont une faible résistivité (10 ohm par carré) et un pouvoir de transmission élevé (75%). Les valeurs de la résistivité et du coefficient de température des films d'or sur de l'oxyde de bismuth sont donnés pour des films de 20-400 Å et pour des températures allant de 15 à 150°C. On montre que le coefficient de température décroit avec l'épaisseur du film, le qui peut être rattaché à une augmentation de la résistivité du dépôt. Le pouvoir de transmission optique et la résistivité des principaux dépôts de chacune des matières citées ci-dessus est donné sous forme de tableau.

INTRODUCTION

Known Methods of Preparation

INTEREST HAS RECENTLY been shown in the production of ' conducting glass ', which is used for resistance heating elements to prevent mist and ice formation on the windows of aircraft, etc. Attempts to make glass electrically conducting by the addition of bulk conducting materials, such as lead oxide, have not been successful, because the products have a greatly reduced optical transmission. The problem, however, has been solved by depositing an adherent conducting layer on the surface of the glass. This layer must have both a high conductivity and high optical trans-

* MS. received June, 1955.

parency, properties which are possessed by some metal oxides.

The preparation of conducting metal oxide films is not a new development, since Baedeker[3] in 1907 prepared conducting layers of cadmium oxide on glass by heat-oxidising sputtered cadmium films in air. However, conducting metal oxide films have recently been deposited directly by :—

(1) Cathodic sputtering of a metal in a glow discharge containing oxygen, this technique being termed *reactive sputtering*, and

(2) Producing a reaction between a metal halide and water vapour.

In this report we are concerned solely with the properties of metal oxide films prepared by cathodic sputtering.

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Vacuum deposition techniques are generally investigated by the writers in order to arrive at satisfactory plant designs. Thus, when techniques for preparing transparent conducting films were reported by Libbey-Owens-Ford, Preston, Helwig and others, these were investigated with the object of determining the type of apparatus best suited for their formation; this was the main subject matter of our article published in the previous issue[1]. During the investigation into deposition techniques much of the published work was repeated and the following account contains information on both original and reported work. It has been published in this form to assist those concerned with the problems of routine film production.

Types of Conducting Films

There are two distinct types of transparent conducting film which have found use as heating elements. These are:—

Metal Oxide Films. The first type of film consists of a semi-conducting metal oxide layer, whose conductivity depends upon either the state of oxidation of the film or the number of impurity centres existing in its crystal structure. Thus the conductivity is not only dependent on the thickness and the physical state of the film, as found with metallic layers, but is also dependent upon imperfections in its structure.

Using one or other of the techniques itemised above, it is possible to deposit electrically conducting films of tin oxide, cadmium oxide, indium oxide and iron oxide, having high optical transparency. Such films may have resistivities ranging from several thousand ohm to 50 ohm per square* with optical transmissions of 75 per cent.

The sputtering conditions affect the conductivity of a deposit because they determine the purity and degree of oxidation of the product. We have previously[1] stressed the need for :—

(1) Careful control of the oxygen content in the sputtering gas.

(2) The elimination of desorbed gas from the discharge atmosphere, and

(3) The temperature control of the cathode and work chamber, etc.

These are known to influence the degree of oxidation of a film. To those we must now add the need for:—

(4) The control of the purity of the cathode metal.

The effect of impurities on the conductivity of metal oxide films is referred to by Aitchison[14] who shows that the effect of impurity may be to enhance or reduce the conductivity of a semi-conductor.

Metal Films. Arising from the interest shown in metal oxide films it has been discovered that the structure of a thin metallic film can often be made to approach that of the compact body if the metal layer is deposited on a metal oxide substrate.[2,16] This second type of deposit also has a high optical transmission and high conductivity, which is a rare combination for thin metal films.

For example, gold films directly deposited on glass at normal temperatures do not develop a connected structure which is conducting until a thickness of about 40 Å is reached, but when deposited on a sputtered bismuth oxide base-coating, gold films become conducting when only 15–20 Å thick. The apparent function of the dielectric layer is to reduce the mobility of the gold atoms on the substrate surface, thereby preventing the formation of an

* The resistance values represent measurements made on a square sample of the surface with electrodes placed on opposite edges. The specific resistance is then equal to the product of the film resistance and its thickness in cm.

agglomerated structure. The conductivity of the gold film can be further improved by the addition of a bismuth oxide top coating which also serves as a protective film.*

Applications

Conducting films, prepared by the foregoing methods have been used as heating elements to prevent the formation of mist or ice on glass windscreens. The resistivity of gold films on bismuth oxide is much less than that of the conducting metal oxide films and gold heating elements can be operated from a power supply with a lower voltage output. It has been claimed that an aircraft window must dissipate 1 kW per sq. ft. to prevent ice formation. Thus a gold coating of 10 ohm per square having a transmission of 75 % could be operated at this power from a 100 volt supply.

Conducting metal oxide films have also been used on instrument windows to prevent the accumulation of electrostatic charges, which affect the meter reading, and in certain electronic devices (*e.g.* some particle accelerators) to prevent the charging of a dielectric surface under electron bombardment.[24] The resistivity of some metal oxide films is stable up to 400°C, and such coatings may prove useful as high power resistors.

Conducting transparent films of cadmium oxide have been used successfully by Preston[9], and Veszi[4] in place of gold films on selenium rectifier photocells; these so-called blue cells are sensitive to light over a wider range of wavelengths, and the overall efficiency of the cell is increased.

Some sputtered oxide films have useful optical properties. For example, bismuth oxide films have been used in optical interferometry as beam splitters, because of their high refractive index and low optical absorption, and iron oxide films have been used for a similar purpose in the infra-red region.

Koenig and Helwig[5] have used sputtered platinum dioxide (PtO_2) films for preparing grain-free replicas of glass surfaces studied by electron microscopy.

PREPARATION OF SPUTTERED FILMS

Cadmium Oxide

The properties of reactively sputtered cadmium oxide films have been investigated by Preston[8], Veszi[3], Helwig[9] and Stuke.[25]

Helwig's report contains a detailed account of the dependence of the electrical conductivity of cadmium oxide films on the sputtering conditions. He showed that the conducting properties were due to the inclusion of excess cadmium atoms in the lattice, and that it was possible for nitrogen to take the place of oxygen atoms, forming a nitride or a mixture of oxide and nitride. He found that the nitride was unstable when heated in air, and that the layer returned to the oxide state accompanied by a decrease in conductivity. The conductivity of pure cadmium oxide films was increased by heating in air, and this was assumed to be due to partial dissociation of the oxide to provide excess cadmium. Helwig was able to relate the partial pressure of the oxygen in the sputtering gas to the conductivity of the deposit, which after heat treatment in air tended to reach a constant and high value of conductivity irrespective of the initial value.

Repeating Helwig's work we have been able to sputter in oxygen-argon mixtures cadmium oxide films on glass which are stable in atmosphere at temperatures up to 400°C.

Experimental Procedure

So many variables exist in sputtering technique that it is essential to investigate them systematically one by one, keeping the rest constant. We started by examining the effect of the state of the discharge (gas density/voltage relationship) upon the condition of the oxide film. Sputtering in pure oxygen from a 45 cm. diameter cadmium-plated cathode with a receiver to cathode spacing of 10 cm, it was found that the highest sputtering rate and most adherent films were produced when the cathode dark space extended almost to the anode surface. The ratio of cathode dark space to positive column was then about 4 : 1. Maintaining these values constant cadmium oxide

* The authors are indebted to Mr. J. S. Preston of the National Physical Laboratory for the useful information he has given them in a private communication describing the properties of a double oxide-metal film combination.

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films were then produced in different gas mixtures and at different support temperatures.

Temperature Effects During and After Deposition

The resistance of the films prepared on a cooled receiver increased with the volume percentage of oxygen in the sputtering gas, but after being heated in air at 400°C for ten minutes all the films tended to have an equally low resistance.

During the initial tests, the jig holding the glass slides was bolted firmly to a water-cooled support, so that the receiving surface was kept cool. By raising the jig a short distance from the cooled surface it was expected that the deposit would be heated by the glow discharge and thus the need for heat treatment in air eliminated. The slides were mounted on a thin mild steel sheet spaced about one centimetre from the cooled anode surface. It was found that the temperature of this support reached 250°C after about 15 minutes sputtering time, and this resulted in a more firmly bonded and harder film with a much lower resistance. This result confirms Helwig's observation that heating cadmium oxide films at reduced pressures increases the conductivity because the oxide begins to dissociate.

The cadmium oxide films deposited on the high temperature support were not sufficiently decomposed to produce the lowest stable resistivity. Given in Table I are the results for six specimens which had been prepared on cooled and uncooled supports and then baked in air. The films were sputtered in a five-minutes period and in a 25 volume per cent. oxygen-argon mixture. Resistance values were recorded during both the heating and the cooling cycle.

Effects of Gas Mixture

A series of cadmium oxide films was deposited using gas mixtures of argon and oxygen in which the oxygen content varied from 100 down to 5 volume per cent. Specimens were prepared in three runs with each gas mixture and the average resistivities before and after baking in air are given in Table II. The films produced in this series were heated during deposition by the glow discharge as described in the above section.

The effect on the resistivity of heat-treating cadmium oxide films in air over the temperature range 20°C – 400°C is shown in Fig. 1. The resistance values are for cadmium oxide films sputtered in a twenty-minute period, *i.e.* having a thickness of about 600 Å*. The curves in Fig. 1 shows that it is possible to complete the transition of the oxide to the highest stable state of conduction during the sputtering process so that heat treatment in air is not required. This is achieved by limiting the oxygen content in

* The thickness of all films was measured by multiple-beam interferometry.[29]

Table I. **The Dependence of the Resistance of Cadmium Oxide Films on the Temperature Attained by the Support During and Subsequent to Coating**

	Baking and Cooling Cycle Resistance in kΩ per Square								
Expt. No.	100°C	150°C	200°C	250°C	300°C	400°C	300°C	200°C	20°C
				Directly Cooled					
1.	100	98	50	15.0	10.0	5.0	4.1	1.7	1.3
2.	60	60	40	14.0	11.0	3.5	3.0	1.5	1.3
3.	58	54	35	10.5	7.0	3.0	2.6	1.5	1.3
				Heated by Discharge					
4.	7.4	6.9	5.8	4.6	5.1	2.6	2.8	1.8	1.4
5.	8.0	7.5	6.8	5.0	4.7	2.8	2.6	1.8	1.4
6.	7.0	6.5	6.0	4.7	4.8	3.5	2.2	1.7	1.3

Note—The above films were sputtered in five minutes at 3 kV and 0.3 mA/cm² from a 45 cm. diameter water-cooled cathode at a receiver distance of 10 cm. using a 25 vol% O_2 -A mixture.

the glow discharge to such an extent that the required number of excess cadmium atoms are included in the lattice while the film is being formed, the receiver temperature rising sufficiently to aid the process. The results given in Table II for the 5 volume per cent. oxygen mixture have been consistently obtained showing that control of the oxygen content is a reliable method for producing highly conducting deposits. This would be the only practical method of depositing highly conducting cadmium oxide films on supports which could not be heated to high temperatures in air *e.g.* selenium rectifier photo-cells.

Resistance Stability with Age

The resistance stability of cadmium oxide films was determined over a period of three months by making regular measurements on films which were electrically heated to 100–150°C, *i.e.* were dissipating some 2 watt per square inch of surface. The average change in the resistance of twelve specimens of different thickness was recorded. These films had already been heat-stabilised at 400°C in air.

During the first week, an ageing effect was observed and the resistance decreased by about 8% to a minimum value. Thereafter the resistance increased slightly until after a further three months the thick films (approximately 600 Å thick) had increased by values up to 1%, and the thin ones (approx. 150 Å thick) by values up to 5%.

The increase of resistance which was observed during life test could have been caused by atmospheric attack of the unprotected films. Cadmium oxide has the disadvantage of being chemically attacked by water vapour and corrosive atmospheres, so that cadmium oxide films must be protected if used in humid atmospheres. This has been achieved in their use on selenium rectifier photocells by coating the cadmium oxide layer with a thin gold film, but it has been shown by Veszi[10] that a transparent coating of lacquer is more effective. In our experiments we have found that a protective layer of bismuth oxide deposited by sputtering is adequate to prevent atmospheric attack. A dielectric film of low refractive index such as magnesium fluoride (MgF_2) might prove more satisfactory however, both for protecting the conducting oxide and reducing its reflectivity.

The adhesion of cadmium oxide films to glass is good, but for rugged use the films must be protected by lacquering or by lamination.

Table II. **The Dependence of the Resistivity and Transmissivity of Cadmium Oxide Films on the Oxygen Content of the Discharge Gas**

Sputtering Time	*Oxygen*	*Resistivity* k Ω *per sq.*		*Optical Transmission at* λ = 5460Å		*Thickness*
		Baking		*Baking*		
Mins.	%	*Before*	*After*	*Before*	*After*	Å.
				%	%	
5	100	18.3	1.96	77	79	—
10	100	9.5	0.545	57	60	—
15	100	5.0	0.315	47	52	—
20	100	4.3	0.288	47	52	426
5	75	9.0	1.490	73	78	—
10	75	6.3	0.440	56	60	—
15	75	4.1	0.297	48	53	—
20	75	3.0	0.238	44	50	509
5	50	7.9	1.690	78	78	—
10	50	5.0	0.427	56	58	—
15	50	4.8	0.290	50	54	—
20	50	2.3	0.180	44	48	543
5	25	9.0	1.150	70	73	—
10	25	1.9	0.436	56	58	—
15	25	1.8	0.182	44	48	—
20	25	1.2	0.134	44	49	570
10	10	0.5	0.260	54	53	—
20	10	0.21	0.126	50	52	509
10	5	1.2	0.520	64	66	—
20	5	0.103	0.110	60	59	644

Note: The above films were sputtered at 3 kV and 0.3 mA/cm. from a 45 cm. diameter cathode at a distance of 10 cm.

Temperature Coefficient of Resistivity

Measurement of the temperature coefficient of resistance on *highly conducting* cadmium oxide films in the temperature range 15°C–150°C usually gave a positive value of the order 0.08 per °C for films about 200 Å thick and up to 0.04 per °C for thicker films about 600 Å thick. Some highly conducting films were produced which showed a reversible decrease in resistance on heating, giving a negative temperature coefficient of the order 0.02 per °C. It

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was difficult to find whether the highly oxidised films of low conductivity possessed a negative temperature coefficient of resistance, since measurement was complicated by the structural changes which accompanied any rise in temperature, giving irreversible increases in conductivity. Cadmium oxide is normally an excess or *n*-type semi-conductor, but it must under certain conditions possess intrinsic conduction properties, or at least it may assume some intermediate state, accounting for the variation in sign of the temperature coefficient of resistance.

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Deposition on Plastics

Reactive sputtering of cadmium oxide on to Perspex was attempted, but this material rapidly outgasses when exposed to a glow discharge, and the surface becomes discoloured due to the decomposition of the plastic under ion bombardment. A similar effect has already been reported for transparent plastics exposed to ionic bombardment prior to vacuum evaporation.[31]

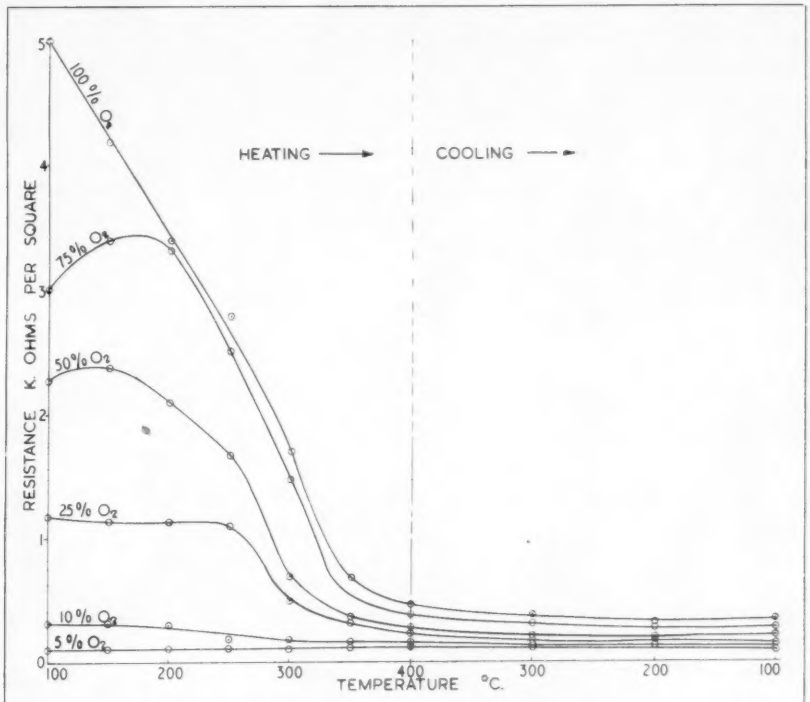

Fig. 1. Resistance of cadmium oxide films upon annealing and cooling. Heating cycle from 100→400→100 °C over 75 minutes. Curves are given for films produced in different oxygen/argon gas mixtures.

Results

The foregoing results for sputtered cadmium oxide films can be generalised as follows[7] :—

(1) The sputtering rate increases with the volume per cent. of argon. The presence of argon in the discharge does not itself influence the conductivity of a given film. Helwig[9] has made the same observation and considers that the argon molecule is too large for inclusion in the cadmium oxide lattice.

(2) Highly conducting films may be produced by carefully limiting the oxygen concentration in the discharge gas during sputtering or by heat treating the deposit in air to promote partial dissociation of the oxide. In both cases the resultant oxide is deficient in oxygen.

(3) Quarter-wavelength films of cadmium oxide about 650 Å thick have a resistance of 100 ohm per square and an optical transmission of some 60% for green light. By increasing the optical thickness to $\lambda/2$ (*i.e.* to about 1,300 Å actual thickness, assuming the refractive index to be 2.1) a maximum transmission for green light of up to 85% may be obtained with resistivities as low as 75 ohm per square.

Tin Oxide

Conducting films of tin oxide produced by spraying stannic chloride solution on to a hot glass surface in the presence of water vapour have been prepared by McMasters[12]. Films produced in this manner have a low resistance of about 50–100 ohm per square with an optical transmission of 80%. Gomer[26] has also prepared conducting tin oxide films by blowing hot air over stannic chloride crystals. By this technique he succeeded in depositing thick films having resistivities as low as 20 ohm per square, but such thick films were granular and possessed a low optical transmission. Preston has shown that conducting tin oxide films can be prepared by reactive sputtering a partially oxidised tin film of brown body colour which becomes conducting when heated in air. This technique was investigated by the authors.

Experimental Procedure

Preliminary observations showed that completely transparent tin oxide films could be sputtered in discharges containing a high proportion of oxygen; these films did not become conducting after baking in air. After sputtering in oxygen there was a change in the colour of the cathode surface due to its oxidation. When large concentrations of argon were used, this oxide was sputtered from the cathode and formed a brown tinted film which changed to a metallic deposit when the oxide layer was completely removed from the cathode. The brown deposit sputtered in pure argon, after pre-oxidation of the cathode, had moderate conductivity and was considered to be a mixture of metal and oxide.

To produce these partially oxidised films, it was necessary to use a 5 volume per cent. oxygen-argon mixture and to reactivate the cathode in a pure oxygen discharge after thirty minutes normal sputtering. Increasing the percentage of oxygen in the gas mixture to maintain an oxide film on the cathode was not effective since thinner highly oxidised non-conducting films were deposited which were not conducting after heat treatment. Pre-oxidation was therefore considered necessary to give the most repeatable and uniform results. However, some experiments were made whose results could not be explained by the previous assumptions. These occurred often after the plant had been out of use for a few days, and it was assumed that gases, particularly nitrogen, occluded on the chamber walls was responsible for the poor results.

Results

Films of tin oxide produced under the best conditions had initially a brown colour and a very high resistance of about 500 kΩ/sq. after 15 minutes sputtering. Upon baking to 400°C the conductivity was increased with an improvement in optical transmission; a final resistivity of 3 kΩ/sq. was obtained for a transmission of 78%. Similar treatment was given to a film of tin oxide produced in a 30-minute sputtering period, and a final resistivity of 1.6 kΩ/sq. was obtained with a transmission of 70%; the films were pale straw coloured. These values were the best obtained for tin oxides; generally the results were variable and although batches produced under similar conditions were uniform in appearance, it was impossible to predict the final resistance after baking. No rise in the conductivity was observed when the sputtered film was deliberately heated by the discharge during its formation as described for cadmium oxide films above. Repeatable results were difficult to obtain with sputtered tin oxides in spite of very careful control of the process conditions.

Aitchison[14] has shown that the presence of impurities has a great effect on the conductivity of chemically prepared tin oxide layers. For example, a low percentage of iron reduces the conductivity of tin oxide, whereas antimony increases it. The 45 cm. diameter cathode used in our tests had been tin electro-plated, and the purity of the metal was unknown. Using a 7.5 cm. cast tin cathode,* in which antimony and iron were the principal impurities, much more repeatable results were obtained at higher values of resistivity and higher optical transmission. For example at an applied potential of 1.5 kV and a current density of 1.5 mA/cm², films of 10 kΩ/sq. and 80% optical transmission were

* Capper Pass tin (total impurity less than 0.003%, antimony and iron being the principal impurities).

obtained after 10 minutes sputtering and subsequent heating in air to 450°C for 10 minutes.

The deliberate control of impurity which may have been necessary to give a higher conductivity was beyond the scope of our programme.

It is doubtful whether tin oxide films produced by reactive sputtering will prove to be suitable for commercial use, because of their high resistance compared with those produced by chemical methods. It is generally believed that the low resistances attained with the vapour reaction method are partly due to migration of impurities from the glass base into the film.

Indium Oxide

Conducting indium oxide films on glass have been prepared by Zunick[15] and by Cusack and Bowness[13] using a vapour reaction method similar to that described above for tin.

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Sputtering Conditions

Indium oxide may be sputtered readily in oxygen, and oxygen-argon mixtures, but it is difficult to obtain the oxide in the conducting state. Indium oxide films sputtered from a water-cooled cathode* (45cm. diameter) showed only the slightest conduction, and it was not until the water-cooling was disconnected from the cathode that reasonably conducting films were produced. The best results were obtained, when the cathode temperature approached the melting point of indium. (Temperature control of the cathode would be necessary for industrial film production). Some films of 300 ohm per square were obtained with a hot cathode and a 50 volume per cent. oxygen-argon mixture, but they were slightly metallic in appearance and their optical transmission was poor. When the oxygen content was decreased to less than about 40 volume per cent. opaque metallic films were sputtered.

It was relatively easy to obtain thick films of non-conducting oxide, pale straw in transmission colour, in a 50 per cent. gas mixture with a cooled cathode, but no conduction was observed even after heat treatment of these films in air.

Results

The best oxide films obtained by sputtering indium, had a resistivity of 2,000 ohm per square with a transmission of 80%. These were produced by sputtering in a 50 volume per cent. gas mixture for 20 minutes from a hot cathode. Heating the film in air decreased the conductivity until the film became non-conducting at about 400°C, which indicated that the excess metal atoms were completely oxidised.

Indium oxide, therefore, showed little promise, and the results were not repeatable.

Iron Oxide

Iron oxides were sputtered in gas mixtures of oxygen and argon, and the proportion of oxygen was progressively reduced until a sputtering atmosphere of pure argon was used. It was found that the residual oxygen in the system was sufficient to give a completely oxidised non-conducting film, having a reddish brown body colour. The oxide sputtering rate was greatest with gas mixtures containing high proportions of argon.

Dushman[28] shows that the dissociation pressure of iron oxides is very low at normal working temperatures, *i.e.*, to dissociate $Fe_2O_3 \rightarrow 2Fe + 1.5\,O_2$ at normal temperatures (20°C) the oxygen pressure would need to be less than 10^{-80} mm Hg. Thus, if formed, the oxide molecule would be highly stable. The rate of oxide formation would, apart from other factors, depend on the oxygen pressure.

Iron oxide can be reduced by hydrogen

$$Fe_2O_3 + 3H_2 \rightarrow 2Fe + 3H_2O$$

providing the ratio of H_2O to H_2 is of the order of 10^{-3} at normal temperatures. Sputtering was therefore carried out in a gas mixture which included hydrogen and with a tray of phosphorus pentoxide drying agent in the sputtering chamber.

Sputtering Conditions and Results

Films were deposited using a cathode 7.5 cm. in diameter separated by 2 cm. from the receiver, at a

* The cathode was prepared by spreading molten indium over a massive copper plate. The indium was of 99.9% purity and obtained from Messrs. Johnson Matthey. Tin was the principal impurity.

potential of 2 kV and a current density of 1.5 mA/sq. cm. in a 30 vol. per cent. hydrogen-argon mixture. Conducting films were produced, and these were pale brown in transmission colour, indicating that some oxidation had still occurred. Films of about 300Å were deposited in 10 minutes, having a resistance of 50-100 kΩ/sq. These films were stable up to about 200°C in air showing reversible changes of resistance with temperature, and a very high negative temperature coefficient of resistance of the order 0.6% per °C. Upon heating above 250°C the resistance increased irreversibly to a value above 200 kΩ/sq. and a second state was reached at about 300°C when the resistance decreased again in the range 300–400°C showing a negative temperature coefficient of resistance of about 0.75% per °C. At higher temperature the films became darker in colour and their resistivities slowly increased to infinity which did not change on cooling.

These changes indicate that the oxide may exist in several different states possibly corresponding to the compositions FeO, Fe_3O_4, Fe_2O_3, the degree of oxidation increasing as the films are heated in air, until the non-conducting completely oxidised state is reached.

Some investigators have observed negative temperature coefficients of resistance in thin films of nickel and iron produced by sputtering, and have attempted to explain this theoretically, on the basis of 'tunnel' effect and granulation. It is likely that they were depositing oxide layers with semi-conducting properties, which possessed a metallic appearance on account of their high reflectivity. Thus Itterbeeck et al.[13] have reported that the resistivity of thin iron films is greatly increased when the films are sputtered instead of evaporated. This increase in resistivity was found although the deposits were sputtered in spectroscopically pure neon from an electrolytic iron cathode. The increased resistance could not be attributed to grain formation since the sputtered films had a much smaller grain size than the evaporated layers. One can only assume that the films were oxide-contaminated.

Gold Films on Bismuth Oxide Substrates

It has recently been claimed by Libbey-Owens-Ford[16] and reported by Preston and Gillham[2], that gold films deposited on to certain metal oxide films have a more continuous structure, resulting in greater light transmission and greater conductivity than gold films deposited directly on to glass.

The manner in which a film condenses on a surface depends on the relation between the adhesion of the film to the surface and cohesive forces in the film itself. Appleyard[17], has propounded a simple theory which predicts the nature to be expected of the structure of a thin film. In brief he said that if the binding forces between the condensed atoms and the substrate were greater than the cohesive forces in the film, then the preferred structure would be a uniform dispersal of the atoms, *i.e.* an homogeneous structure. If, on the other hand, the binding forces at the interface and between the atoms of the film are in reverse relationship then the preferred structure would be granular. The substrate temperature may need to be raised for the condensed atoms to obtain sufficient mobility to move into an agglomerated structure.

An example of this can be found in everyday life. If one breathes on a surface of glass, contaminated by a thin layer of grease, the breath deposit forms a series of discrete water droplets which scatter light. If, however, one thoroughly cleans the surface of the glass by flaming it or by ion bombardment at low pressures then, when it is breathed upon, a breath figure cannot be observed. The presence of a condensed moisture film can however be detected by a reduction in the reflection from the glass surface and this condition has been termed a *black breath figure*. The explanation for this is simple. Water molecules have a high affinity to clean glass surfaces and thus when condensed form a continuous non-granular layer. The refractive index of water is lower than that of glass and this is the reason for the decrease in the surface reflection. When the water layer evaporates and is of interference thickness reflection colours can be observed similar to those of anti-reflection coating. A similar example to the black breath figure is sometimes found with vacuum deposited layers. Thus in the preceding example, gold layers deposited directly on glass have a granular structure due to poor adhesion of their atoms even to

clean glass surfaces, whereas if the glass surface is precoated with bismuth oxide the gold films have a more homogeneous structure as shown by their transparency and high electrical conductivity.

Sputtering Conditions

Bismuth oxide is freely sputtered in an oxygen discharge, and the sputtering rate may be increased by using a gas mixture of 50 volume per cent. oxygen and argon. If higher proportions of argon are used, opaque metallic or semi-metallic films are deposited.

Evaporated and sputtered gold films were deposited on to bismuth oxide films on glass and on to plain glass slides with the following general results:—

(1) Sputtering in pure argon produced more firmly bonded gold films on glass than evaporation and was therefore preferred.

(2) Conductivity of the gold film (on the oxide layer) for a given thickness was comparable whether prepared by evaporation or by sputtering.*

(3) When deposited on bismuth oxide, the conductivity of the gold coating was greatly increased, especially for the thinnest gold films (60 Å thick) and the transmission colour was straw instead of green-blue.

(4) The conductivity was further improved by depositing a second layer of oxide on top of the gold and heating the combined layers to 300°C – 350°C. Heating above 350°C destroys the conductivity of thin gold films less than about 80 Å thick.

(5) A minimum thickness of 80 – 90Å is required for each oxide layer to give the best results. Thereafter it depends on the choice of optical thickness to obtain the highest transmission.

(6) During life test over a period of 3 months an ageing effect was observed. The conductivity slowly increased by as much as 30%, afterwards remaining constant to within 1%.

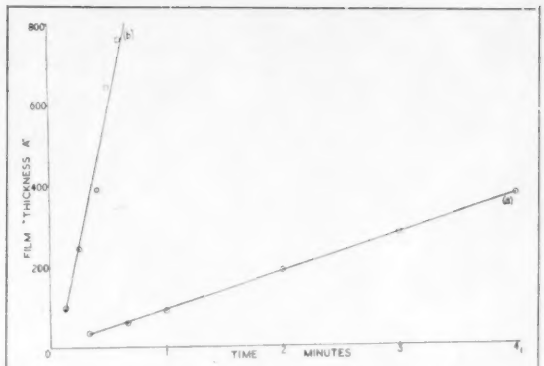

Fig. 2. Sputtering rate of gold. (a) Current density—0.3 mA/cm² at 3kV. Cathode diameter —45 cm. Receiver distance—7.5 cm. (b) Current density—1.5 mA/cm² at 3 kV. Cathode diameter—20 cm. Receiver distance ~3 cm.

Dependence of Resistivity on Deposition Rate

Using a current density of 0.3 mA/sq.cm. at a

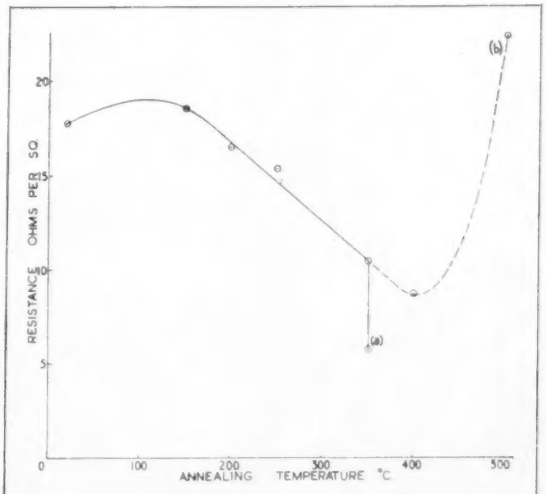

Fig. 3. Resistance changes in a typical gold-bismuth oxide layer on annealing. (a) Resistance decreases on annealing at 350°C for 10 minutes. (b) Film is destroyed when heated above 350°C.

* It should be noted that gold films deposited by vacuum evaporation from tungsten filaments at a high pressure of 0.1 to 1 micron Hg have a fine grain structure making them useful for replica shadowing in electron microscopy, whereas gold films deposited at low pressures (0.001 micron Hg) have a granular structure. Birks[18] found that the difference in structure arose from the presence of tungsten oxide in the deposit, because a volatile oxide was formed on the filament surface at high gas pressure. Such compact gold films could have a greater resistivity due to this impurity.

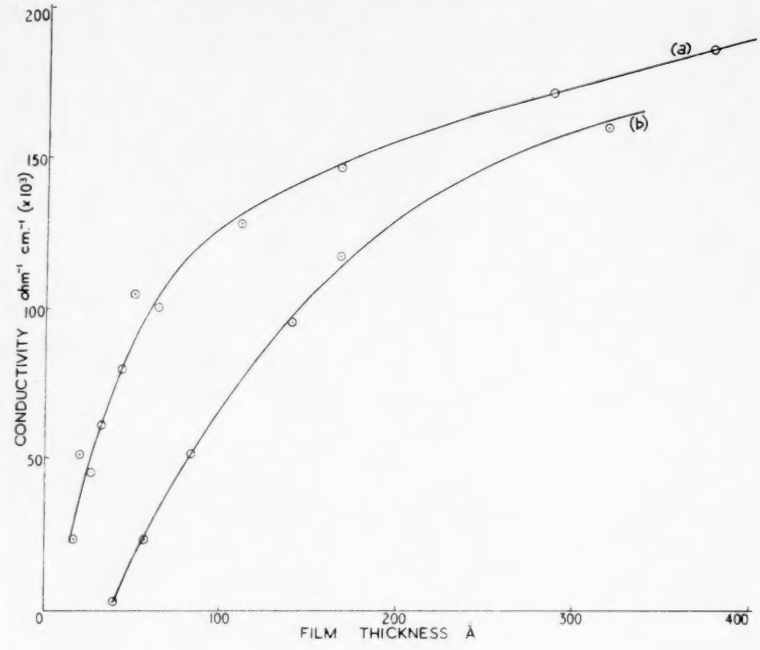

Fig. 4. Variation in conductivity of thin gold films with thickness. (a) Deposited on Bi_2O_3 and annealed at 300°C. (b) Deposited directly on glass.

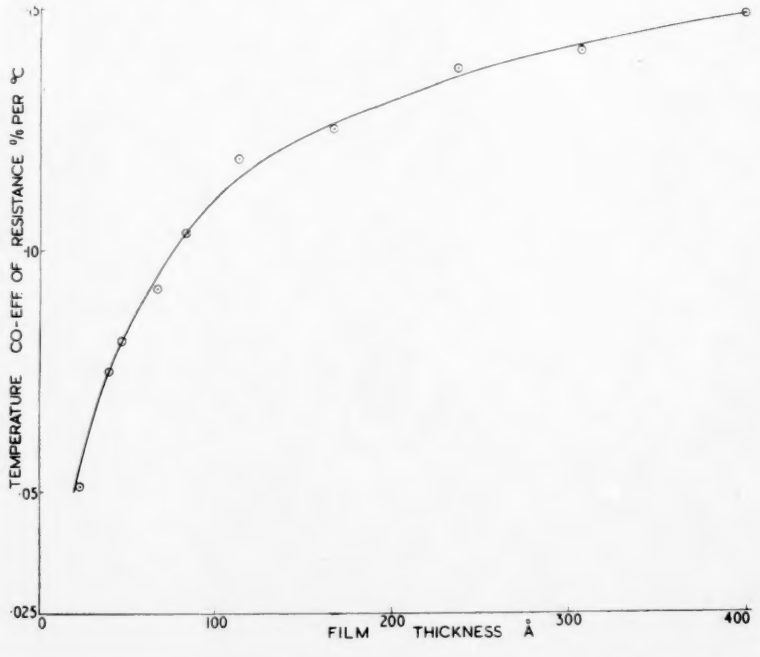

Fig. 6. Variation of temperature coefficient of resistance of gold/bismuth oxide layers with thickness.

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potential of 3 kV with a 45 cm. diameter cathode, a film of bismuth oxide 90Å thick was sputtered in 2 minutes, and a suitable gold film (80Å) was obtained after about 1 minute under the same conditions. The total sputtering time to produce a composite film of resistance 20-24 ohm per square with optical transmission of 72% was just over five minutes. After heating this film in air at 350°C for ten minutes, the resistance was reduced to 8–10 ohm per square and the transmission increased to 75–78%.

Further tests were made using a cathode only 20 cm. diameter and a spacing of 2.5–3 cm. with a current density of 1.5 mA/sq. cm. and a voltage of 3 kV. The power input per square centimetre of cathode was increased with this arrangement by a factor of five. In Fig. 2 the film thickness has been plotted against sputtering time for the two different current densities, and the graph shows that the rate is approximately linear, but for very short sputtering periods at a high current density the results may be inaccurate because several seconds may elapse before the discharge becomes stable. The smaller cathode was not water-cooled and this could account partly for the greatly increased sputtering rate of 12 times which was observed at the higher current density.

When gold was deposited on bismuth oxide at the higher sputtering rate, lower resistivities of 4 – 6 ohm per square were obtained for a transmission of 75%. It was assumed that at the faster deposition rate a more compact layer was formed. Sennett and Scott[23] and Levinstein[27] have shown that increasing the rate of deposition of evaporated layers favours the formation of a compact deposit.

Decay of Resistivity with Rise in Temperature

Vand[19] has studied the changes in resistivity of a metal film which may occur after its deposition. The resistivity of a freshly deposited metal film usually shows a decrease of resistivity as the film ages. Such resistance changes occur more rapidly and fall to a stable value as the temperature at the film is raised. At a very high temperature the resistivity may suddenly rise to infinity due to grain growth or stresses in the film disrupting the coating. Vand attributed the irreversible decrease in resistivity to the decay of lattice defects or distortions in the film. He suggested that lattice deficiences and excesses existed so close together that a small amount of energy would be required to unite them, and that discrete levels existed on account of the different types of defect which were involved. It would seem that a definite temperature is required to remove the defect at each level and that a period of heating at each temperature level is needed for the transition to be completed. For gold films deposited at room temperature only one such level has been noted, *i.e.*, at about 250 – 300°C, but Wilkinson, depositing gold films at liquid air temperatures, found three levels in the temperature range 90 – 273°K. (The resistance-thickness curve given by Wilkinson[20] shows that his films possessed the high resistivity of granular gold films condensed on glass without a cementing layer).

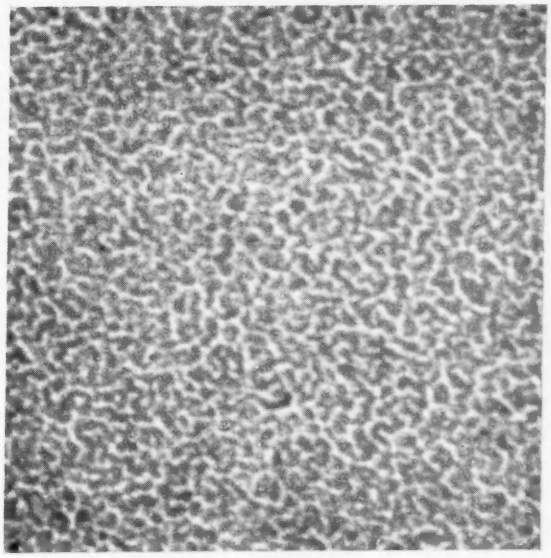

Fig. 5. Thin gold layer on bismuth oxide (both evaporated). Magnification 100,000x.

The decay of resistivity of a typical gold on bismuth oxide layer is given in Fig. 3, showing the destruction

of the film at temperatures above 400°C.

Upon heating a film after deposition at room temperature, two changes may occur. Firstly, the disordered lattice is rearranged, and secondly, the occluded gas is released. The resistance falls to a fairly constant value (subject to external atmospheric effects) after which no large irreversible changes are observed; the film is then assumed to be in its most ordered state for a given method of formation. The rate of decay of resistivity depends upon the heat treatment which is applied, whereas the lowest resistivity attained depends also upon the processing conditions during deposition. If the heating of the film is prolonged at a high temperature other undesirable factors are introduced, even when the heating is carried out *in vacuo*, and granulation or re-crystallisation occurs.

The changes occurring in the composite layers may now be partly understood. Heating to 350°C improves the conductivity by up to four times, but after further heating at lower temperatures for prolonged periods, a smaller increase in conductivity was measured showing that re-ordering of the lattice was still taking place. Films of gold on bismuth oxide have a pale straw transmission colour, instead of the blue-green colour of thin gold films deposited directly on glass. When the composite films are heated above 350 – 400°C (depending upon the thickness) the conductivity rapidly decreases and the transmission colour reverts to blue-green showing that the uniform layer has been destroyed and granulation of the gold has occurred.

Resistivity - Film Thickness Relationship

In Fig. 4(a) the conductivity of gold films sandwiched between bismuth oxide coatings is plotted against film thickness. The films were deposited at 3 kV and 0.3 mA/cm.^{2}. Measurement of the conductivity was made after annealing the coatings at 300°C for 10 minutes with the total heating and cooling cycle taking 1½ hours.

For gold films 80–100 Å thick, the conductivity increased 2–3 times by depositing on the bismuth oxide substrate, and for this order of thickness films of resistance 8–10 ohm per square are produced with an optical transmission of up to 78%.

Curve 4(b) gives the results for gold films directly deposited on glass and a comparison of the two curves shows the effect of the bismuth oxide in reducing granulation. The gold films on glass were not annealed after deposition.

Measurement of the resistivity of thin metallic films has been made by Lovell[6] and Appleyard[17]. They developed a theory to explain the very high resistivity of such films in terms of boundary limitation to the free movement of conduction electrons in the layer. Their theory was in turn extended by Fuchs[7] and Sondheimer[8].

There is some uncertainty whether the films studied by previous workers were truly homogeneous layers. It has been suggested by Preston and Gillham that gold films on bismuth oxide might prove more reliable for this purpose. The coatings studied here had resistivities of the order 2 – 3 times that of the bulk metal. Whether this was due to grain growth or mean free path limitation is uncertain. Electron micrographs (see Fig. 5) indicate that there is still some granulation when the gold is deposited on bismuth oxide.

Conductivity and Temperature Coefficient of Resistance

The temperature coefficient of resistance α of gold films sandwiched between bismuth oxide coatings was measured over the range 15–150°C and the results are given in Fig. 6, which shows that the temperature coefficient decreases with film thickness.

Thin metallic films exhibit irreversible changes in resistance with a rise in temperature unless they have been already thoroughly stabilised by heating to promote structure changes. Thus it is necessary to determine α over a complete temperature cycle in order to find whether any irreversible changes occur during the temperature rise.

Coated slides were immersed in an oil bath which was electrically heated and stirred, and the resistance measured with a Wheatstone bridge. The resistance could be measured with an estimated accuracy of $\pm 0.1\%$ and the temperature determined to within ± 0.25°C. The temperature was measured over the

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range 15–150°C using a mercury thermometer. For each determination the percentage change in resistance was recorded against temperature over the complete cycle.

Comparison with the conductivity curve shows the temperature coefficient curve to be almost identical in shape. A direct relationship can be established for all except very low values of film thickness t, and approximately

$$\frac{\alpha}{\alpha_F} = \frac{\sigma}{\sigma_F} \qquad (1)$$

where α and σ are bulk values of temperature coefficient of resistance and conductivity respectively. The suffix F denotes the thin film modification.

The values of α/α_F and σ/σ_F over a range of thicknesses are given in Table III, showing that there is good agreement for the thicker films. It is interesting to note that Reynolds and Stilwell[21] have found that the product of specific resistance and temperature coefficient of resistance is a constant for evaporated silver and copper films, the resistivities of which increased as the thickness decreased.

It would be incorrect to regard the above as a rigid relationship, since α is not a fundamental constant for a conductor, and its value varies considerably with temperature, but over a restricted temperature range in which α is defined by

$$\alpha = \frac{I}{\rho_\theta} \frac{d\rho}{d\theta} \qquad (2)$$

where ρ_θ is the resistivity at temperature θ, it can be shown that Equ. 1 holds approximately.

Matthiessons Rule states that the resistivity of a conductor ρ may be regarded as the sum of two parts, the residual resistance (at 0°K) due to lattice defects and impurities ρ_L, and the resistance due to thermal oscillations of the atoms ρ_T. When the conductor is a thin film, the thickness of which is less than the mean free path of the electrons the addition of a third resistance term ρ_B is considered necessary by Vand[19] and Wilkinson[20] and others, because the electrons are considered to lose energy by inelastic collisions at the film boundaries so that

$$\rho = \rho_B + \rho_L + \rho_T \qquad (3)$$

In a bulk conductor at normal temperatures, ρ_T is usually very much greater than the residual resistivity, but when the conductor is a thin film, $\rho_B + \rho_L$ may increase considerably, due to boundary limitation and granulation. Over a restricted temperature range we may regard ρ_B and ρ_L to be independent of temperature (provided that the structure of the film is not changed), and therefore any temperature changes occurring in a thin film will have less effect upon the total resistivity, *i.e.*, the temperature coefficient of resistance is reduced. This reduction can be related to the decrease in conductivity, since by definition

Table III. **Comparison of the Temperature Coefficient of Resistance and Conductivity of Gold Films of Different Thicknesses**

t (in Å)	α/α_F	σ/σ_F	t (in Å)	α/α_F	σ/σ_F
400	2.28	2.16	100	3.09	3.18
300	2.39	2.38	80	3.37	3.48
250	2.48	2.58	60	3.74	4.10
200	2.59	2.74	50	4.05	4.61
150	2.76	2.90	40	4.48	5.48
			30	5.23	7.13

t = Film Thickness
σ = Conductivity
α = Temperature Coefficient of Resistance
F denotes the Thin Film Value

$$\alpha = \frac{I}{\rho} \cdot \frac{d\rho_T}{dT} \quad \text{(in bulk conductors)}$$

and

$$\alpha_F = \frac{I}{\rho_F} \cdot \frac{d\rho_T}{dT} \quad \text{(in thin films)}$$

The differential coefficient is the same in each case, because the *intrinsic* rate of increase of resistance is independent of boundary effects, granulation or lattice distortions, and therefore

$$\frac{\alpha}{\alpha_F} = \frac{\rho_F}{\rho} = \frac{\sigma}{\sigma_F}$$

Table IV. **Comparison of the Resistivity and Transmissivity of Sputtered Films**

Material	*Average Results*			*Sputtering Conditions*				
	Optical Transmission % at λ = 5460Å	*Resistance ohm/sq.*	*Temperature Coefficient of Resistance % per °C*	*Applied Voltage kV*	*Current Density mA/cm²*	*Sputtering Rate Å/min.*	*Total Sputtering Time min.*	*Gas Mixture*
Bismuth Oxide	92	∞	—	3	0.3*	45	2	50% Argon 50% Oxygen
Gold	65	50—100	(pos.) 0.15	3 3	0.3* 1.5†	90 1,100	1 0.08	100% Argon
Combined Layers of Gold—Bismuth Oxide	75 75	10 6	(pos.) 0.1	3 3	0.3* 1.5†	as above	5 1	as above
Tin Oxide	78	3,000	(neg.) 0.1	3	0.3*	25	15	95% Argon 5% Oxygen with cathode activation
Cadmium Oxide (λ/4)	60	100	(pos.) 0.02 or to (neg.) 0.06	3	0.3*	30	20	95% Argon
Cadmium Oxide (λ/2)	85	75	(pos.) 0.02 or to (neg.) 0.06	1.5–2	1.5‡	80	15	5% Oxygen
Indium Oxide	72	1,500	—	3	0.3*	20	20	50% Argon 50% Oxygen
Iron Oxide	75	50,000	(neg.) 0.5	1.5–2	1.5‡	30	10	30% Hydrogen 70% Argon with Phosphorous Pentoxide drying agent

* 45 cm. dia. cathode, water-cooled † 20 cm. dia. cathode, uncooled ‡ 7.5 cm. dia. cathode, uncooled

The deviations which were observed for very thin films could have been due to inaccuracy in thickness measurements or to the invalidity of our assumptions where the film was only ten or twenty atomic layers thick, and where small changes in temperature could have produced changes in the structure.

Results

Gold-bismuth oxide layers with a resistance of 8–10 ohm per square and optical transmission of 75–80% (thickness of gold 80–90Å) have a temperature coefficient of resistance of about 0.1% per °C. They may be produced by sputtering three layers to form a 'sandwich' film. A current density of 0.3 mA/sq. cm. at a potential of about 3–6 kV is adequate for rapid deposition using a cathode of 45 cm. diameter. The total sputtering time to produce this composite layer is under the above conditions about 5 minutes, but unless some automatic change-over of cathodes is provided, the total processing would necessitate three pump-down periods.

Films of resistance 4–6 ohm per square and 75% transmission are obtained when sputtering is carried out using a current density of 1.5 mA/sq. cm. at 3 kV. Such high current densities might prove difficult to use under industrial conditions, because the processing times are about 10 seconds for the gold layer and about 20 seconds for each oxide layer, and it is not

easy to bring the discharge to a stable condition within a few seconds.

GENERAL CONCLUSIONS

The most important results of our investigations into the properties of oxide films are summarised in the Table IV.

The composite gold-bismuth oxide layers were found to have good stability, and should be eminently suitable for making heated windows operated from low-voltage power supplies.

Of the semi-conducting oxides which have been investigated, cadmium oxide gave the highest values of conductivity and most uniformly repeatable results, but the possible use of the other oxides should not be discounted. Tin oxide may prove useful for anti-static coating where a high conductivity is not required, but where durability and freedom from absorption colour are essential.

Acknowledgements are made to Mr. J. S. Preston for the useful information he has given us on the properties of conducting oxide and gold films and to Mr. A. S. D. Barrett, technical director of Edwards High Vacuum Ltd., for permission to publish this report.

The electron micrograph is reproduced by kind permission of the A.E.I. Research Laboratory, Aldermaston, Berks.

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the effect of the substrate temperature on THE CONDENSATION COEFFICIENT OF EVAPORATED ANTIMONY, GOLD AND SILVER

F. M. Devienne, Dr. Sc. Phys.

*Mediterranean Laboratory of Thermodynamic Research, Nice, France.**

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Summary

AFTER A BRIEF reference to previous work on the effect of the thickness of the deposit on the condensation coefficient of antimony and cadmium evaporated onto supports maintained at normal temperature, the author reports the results of a recent investigation into the effect of the change in substrate temperature on the condensation coefficient of antimony and gold. These experiments were conducted with radio-active isotopes and confirm the theoretical assumption that the condensation coefficient decreases as the substrate temperature increases. In the case of antimony the decrease is considerable, even if the substrate temperature has been raised only slightly. In the case of gold the condensation coefficient hardly decreases at all and remains at a value near unity as long as the substrate temperature is not higher than 100°C. But when the temperature rises beyond 300°C the coefficient of gold decreases appreciably as long as the thickness of the deposit is small. The condensation coefficient depends not only on the thickness of the film but also on the nature of the film support as was to be expected from the results of the previous experiments with antimony and cadmium at normal substrate temperature. Experiments with silver indicate a value near unity as long as the film is thin and deposited on substrates maintained at normal temperature. Here, as in the case of gold, the condensation coefficient appears to decrease when the substrate temperature rises beyond 300°C. However, as the radio-activity of silver is poor no precise numerical values can be given.

Sommaire

APRÈS AVOIR RAPPELÉ les résultats publiés précédemment sur la variation du facteur de condensation en fonction de l'épaisseur pour l'antimoine et le cadmium à la temperature ordinaire, l'auteur donne des résultats sur les variations des facteurs de condensation de l'antimoine et de l'or en fonction de la température. Ces résultats, obtenus au moyen des isotopes radioactifs, confirment ce que l'on était en droit d'attendre du point de vue théorique, à savoir : que le facteur de condensation diminue en fonction de la température du support. Dans le cas de l'antimoine, la diminution est assez grande lorsqu'on élève relativement peu la température. Au contraire, dans le cas de l'or, le facteur de condensation ne diminue pratiquement pas tant que la température ne dépasse pas 100 degrés C, il est alors voisin de l'unité. Par contre, lorsque la température dépasse 300 degrés C, le facteur de condensation diminue assez fortement, lorsque l'épaisseur est très faible. Sa valeur dépend, non seulement de l'épaisseur de la couche mince formée, mais également de la nature du support. Résultats qui généralisent ceux que l'on avait obtenus pour l'antimoine et le cadmium à la température ordinaire. Dans le cas de l'argent, des expériences ont été effectuées. Elles indiquent un facteur de condensation voisin de l'unité pour des épaisseurs relativement faibles à la température ordinaire. Il semble, comme pour l'or, que ce facteur de condensation diminue avec la température de la surface condensatrice lorsque celle-ci dépasse 300 degrés C. Mais étant donné la faible radioactivité de l'argent, il n'est pas possible de donner des valeurs numériques suffisamment certaines.

RESULTS OF PREVIOUS WORK

The author and his associates have shown in previous publications[1,2,3,4] that generally the condensation coefficient of a molecular beam, *i.e.*, the ratio between the number of the molecules condensed on a surface and the total number of molecules impinging on it—is neither equal to unity nor zero. It has also been stated in these publications

*MS. received April, 1955

that the value of the condensation coefficient was found to depend on the thickness of the deposit, increasing as the thickness grows larger.

Initially, the nature of the plate on which condensation takes place has a considerable influence. But this influence diminishes as the deposit continues to build up. A series of figures for the value of the condensation coefficient under these conditions has already been published[7] and is quoted below. All the previous experiments with antimony and cadmium were carried out at normal substrate temperature and it should be mentioned here that the results for antimony were more complete than those for cadmium. This is due to the fact that the specific activity of antimony is higher and lasts longer than that of cadmium. Experiments with radio-active gold were also undertaken at the time but the results are not collated yet.

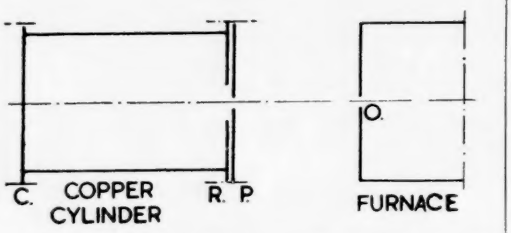

Fig. 1. Diagram of the experimental arrangement used by the author.
O = Orifice in the furnace; C = Condensing plate.

We have now conducted a new series of experiments —the subject of the present article—investigating firstly the characteristics of the condensation coefficient of gold and silver if deposited on substrates maintained at normal temperatures and secondly the effect of a change in substrate temperature on the value of the condensation coefficient.

EXPERIMENTAL EQUIPMENT

There is no need to give a detailed description of the apparatus employed in the experiments. It will suffice to mention that the molecular beam was obtained in a conventional manner by heating the radio-active material in an electric furnace, various types of which have been used. The furnace was situated in a metal bell jar which could be evacuated. Evaporation was carried out at a pressure level of 10^{-5} mm. Hg. The pressure was measured either by a hot ionisation gauge or, where higher pressures were employed, by a Penning-type ionisation gauge.

Six different bell jars were available for the experiments ranging in size from 0.3 to 1.5 m. diameter. The bell jars were fitted with a large number of vacuum-sealed rods passing through the chamber walls, varying in number from 10 to 42. With the help of these rods temperatures could be measured in all places of interest to the experimenter, in particular in the furnace and on the condensing plate. In addition, the rods were used for the manipulation of iris diaphragms and generally for the moving of parts inside the evacuated chamber. Naturally, they also served as lead-in electrodes for the supply to the heater elements of the furnace and the plates on which the beam was to be condensed.

The procedure of determining the condensation factor was as follows :—

The molecular beam emerged from the orifice O of the furnace, see Fig. 1, passed through two plates, P and R, clamped together and provided with 1-2 mm. diameter holes in the centre, and penetrated into a metal cylinder, 20-30 mm. long and 12.5 mm. in diameter. The plate P merely served as a shield for plate R to protect the latter against the vapour travelling in the beam. The molecules of the beam, entering the cylinder, impinged on the base of the cylinder, formed by a detachable plate C and made from copper or some other material. Those molecules which did not come to rest on plate C and were re-emitted, finally condensed on the cylinder walls or on Plate R. The radio-activity of these faces could be determined after completion of an experimental run by dismantling the cylinder assembly. The orifice O of the furnace had a diameter of 1-2 mm. and was placed at a distance from plate C which ensured that the cross-sectional area of the molecular

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beam hitting plate C was well within the cross-sectional area of the cylinder.

It is clear from this description that the condensation coefficient is determined by the ratio of the intensity of the radio-activity of the deposit on plate C and the sum of the radio-activities of all deposits in the cylinder, *i.e.*, on plate C, on the cylinder wall and on plate R. The method of measurement employed excluded all effects of back scattering and those peculiar to the geometry of the counting apparatus. The alignment of the experimental set-up was checked after each run by preparing autoradiographs of the deposit on plate C.

The heating of the condensing plates was effected in two ways: (*a*) A resistance heater element was placed underneath the condensing plate, insulated from it by a very thin sheet of mica and (*b*) a Pyrex or quartz tube, carrying a heating coil of Nichrome or tungsten on the outside, was placed in a position surrounding the condensing plate. The latter arrangement, shown in Fig. 2, had the advantage that the heat produced on the condensing plate was more uniform. On the

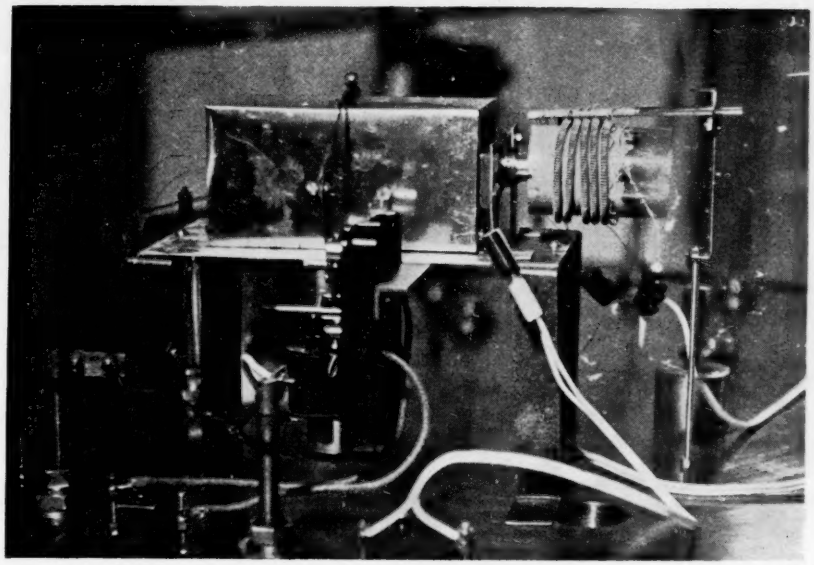
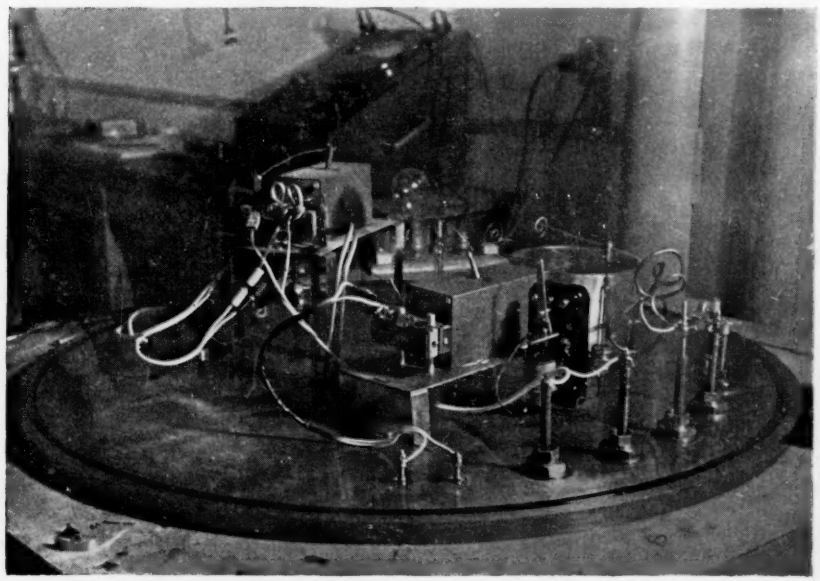


Fig. 2. Furnace assembly, showing also heating system for the condensing plate.

Fig. 3. Baseplate of one of the bell jars fitted with two furnaces.



other hand, it required more vigorous cooling of the cylinder wall and the plate R.

Cooling of these parts was necessary, for instance, in the experiments with gold, where particularly high oven temperatures were needed. The cooling means was water, passed through a helical coil surrounding the cylinder. As a result the temperature of the cylinder wall and the plate R rose only slightly, some 4-5°C, although the furnace was operated at temperatures as high as 1,400°C.

The condensing plates were thoroughly degassed before each experimental run. The temperatures employed for degassing ranged from 300-500°C, according to the nature of the plate material.

THE CONDENSATION COEFFICIENT OF ANTIMONY

As a basis, Table I shows the numerical values of the condensation coefficient of antimony as determined during deposition on substrates maintained at normal temperatures.

Table I. **The Condensation Coefficient of Antimony**

(*a*) Deposited on a Copper Plate Held at 20°C

Thickness of Deposit	*Value of Condensation Coefficient*	*Thickness of Deposit*	*Value of Condensation Coefficient*
1.9 Å	0.401	22.7 Å	0.471
2.2 Å	0.417	31.5 Å	0.580
4.9 Å	0.456	39.6 Å	0.608
42.1 Å	0.638	209.7 Å	0.771
42.4 Å	0.618	340.7 Å	0.763
94.3 Å	0.640	437.8 Å	0.772

(*b*) Deposited on a Glass Plate held at 20°C

Thickness of Deposit	*Value of Condensation Coefficient*	*Thickness of Deposit*	*Value of Condensation Coefficient*
1.3 Å	0.311	31.7 Å	0.464
2.6 Å	0.307	55.0 Å	0.562
12.4 Å	0.322	132.2 Å	0.768
13.6 Å	0.400	497.5 Å	0.829

In the present experiments the temperature of the condensing plate was raised and as a result the values of the condensation coefficient diminished. This is clearly demonstrated by the data shown in Table II which gives the condensation coefficient of antimony deposited on a copper plate held at 170°C and on a glass plate held at approximately 120°C. Incidentally, it should be noted from this data that the nature of the substrate material has an appreciable influence on the value of the condensation coefficient. This aspect is taken into account below.

The figures in Table II show that the condensation coefficient of antimony increases as the thickness of the deposit grows and conform in this respect to the results obtained at normal substrate temperature. But for any given thickness the condensation factor measured on the heated substrates is distinctly lower than the respective value obtained at normal substrate temperature. In the case of glass as a substrate the temperatures employed were generally lower but here again the values of the condensation coefficient measured on the hot condensing plate were proportionally lower. It should be mentioned in this connection that the values obtained on glass were less easily reproducible than those obtained on copper,

Table II. **The Condensation Coefficient of Antimony**

(*a*) Deposited on a Copper Plate Held at 170°C

Thickness of Deposit	*Value of Condensation Coefficient*	*Thickness of Deposit*	*Value of Condensation Coefficient*
0.27 Å	0.24	5.6 Å	0.349
0.63 Å	0.291	12.2 Å	0.52
4.55 Å	0.325	132.2 Å	0.768

(*b*) Deposited on a Glass Plate Held at 120°C

Thickness of Deposit	*Value of Condensation Coefficient*	*Thickness of Deposit*	*Value of Condensation Coefficient*
0.106 Å	0.046	1.14 Å	0.053
0.47 Å	0.0124	108.7 Å	0.122

especially at comparatively high temperatures.

Additional experiments were carried out at temperatures still higher than those shown in the tables. Their results confirm the relationship already

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established, *i.e.* that the values of the condensation coefficient decrease as the temperatures increase.

THE CONDENSATION COEFFICIENT OF GOLD

At normal substrate temperatures the condensation coefficient of gold is near unity. The actual values vary between 0.90 and 0.99. Within this range the values increase as the thickness of the deposit grows. The nature of the substrate material has no marked influence.

The experiments with heated condensing plates showed that a temperature rise not exceeding 300°C has little effect. Measurement of the condensation coefficient in that range produces values almost identical to those obtained at normal substrate temperature. But if the temperatures are higher the value of the condensation coefficient decreases. Just above 300°C the change is still small if a copper substrate is used, but the change is appreciable, if the support is of glass or aluminium. For example, in the case of copper a condensation coefficient of 0.85 was measured for thicknesses of the deposit of the order of 1.5 Å when the plate temperature had reached 350°C. On the other hand, if the plate was glass, the coefficient was found to be 0.5 for a deposit 0.92 Å thick at a plate temperature of 360°C. Moreover, its value is still lower at a smaller thickness of the deposit. Measuring the condensation coefficient on a glass plate heated to the same temperature as before and covered with a deposit of only 0.16 Å thickness, gave a value of 0.2. Condensing gold on aluminium results in a large change of the condensation coefficient as soon as the plate temperature is raised. Thus, a value of 0.715 was established for a deposit approximately 1.7 Å thick at a plate temperature of 320°C. For a plate temperature of 345°C the value was approximately 0.37.

THE CONDENSATION COEFFICIENT OF SILVER

As the specific activity of silver is low it has not been possible to obtain very precise results in the experiments with silver. However, repeated experiments with substrates at normal temperatures indicated that the condensation coefficient is not precisely equal to unity, but fairly close to this value, ranging between 0.9 and 1, as in the case of gold. Here again, when the temperature of the condensing plate is raised, the coefficient diminishes but, as before, it appears that the effect is only great enough to be noticeable at temperatures above 300°C. As the measurements lacked the desired precision due to the low intensity of the radiation no evidence could be obtained on the influence of the nature of the support material.

Since this article was first compiled Ling Yang, M.T. Simnad and G. M. Poundi have published the results of a study of the condensation coefficient of silver carried out with the help of radio-active metal using silver, gold, platinum, nickel and glass as support materials—heating the condensing plates to 192°C in every case. They found the following values for the condensation coefficient (in the order of the support materials given) : 1, 0.99, 0.86, 0.64 and 0.31.

CONCLUSIONS

From the above it can be seen that the value of the condensation coefficient depends on the temperature of the substrate. It decreases as the substrate temperature increases. This condition seems to apply generally but in the case of antimony the changes are more pronounced than in the case of gold and silver. These differences may originate from the fact that the vapour pressures of the deposited material differ at a given temperature of the support.

The problem of the changing values of the condensation coefficient is very complex. Beside the influence of the temperature and the properties of the support material other factors play a part such as the condition of the receiving surface and the temperature of the molecular beam.

The actual mechanism of condensation is certainly very intricate. The simplest theoretical approach to an analysis is the *a priori* assumption that one of two possible processes will occur during deposition, (*a*) the molecules of the beam condense on the support

or (*b*) the molecules are re-emitted from the support. However, this premise does not explain satisfactorily the changes of the condensation coefficient caused by changes in the substrate temperature. According to this theory it appears logical that the condition which leads to the re-emission of a molecule is based on the nature of the substrate material or that of the deposit formed on the substrate in the process. But it is not equally clear, how a comparatively small rise in substrate temperature could affect the conditions as much as is demonstrated by the experiments.

The first step towards a methodic study of the mechanism of condensation has just been made as a result of research carried out recently in the laboratory of the author. During experiments evaporating inactive material onto a thin radio-active deposit it was established that a certain number of molecules in the deposit serving as the substrate were ejected during the deposition of fresh material. This indicates that in the course of condensation from the vapour beam substitution of molecules in the deposited material by molecules impinging subsequently on the surface of the condensed layer may occur to a varying degree.

If it is taken into account that a rise in the temperature of the plate supporting the deposit weakens the forces of attraction between the molecules in the deposit, it will be understandable that, in these conditions, the molecules in the condensed layer are comparatively easily ejected under the impact of freshly impinging molecules. At the same time it will be realised that other factors contribute to the changes of the condensation factor, for instance, the temperature of the molecular beam.

A number of investigations are now in progress in the laboratories of the author aiming, (*a*) at establishing what other conditions have an influence on the condensation coefficient and (*b*) at exploring the mechanism of exchange between the atoms and molecules condensed on the surface and others impinging on it.

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ISOLATION VALVES for vacuum systems

J. Yarwood, M.Sc., F.Inst.P. — *Head of the Mathematics and Physics Department, The Polytechnic, London, W.**

Summary

THE CHIEF TYPES of vacuum valve used for isolating one part of a vacuum system from another are classified and many of the designs published in the last twenty years are reviewed. The requirements of a vacuum valve are considered and criticisms and comments made regarding existing types of valve. Vacuum valves developed from existing valves as used in routine engineering practice as well as valves designed specifically for low pressure work are described. An account follows of recent work on valves for ultra-high vacuum systems. Possibilities for future designs, chiefly of the type of valve which can be degassed by baking, are briefly discussed.

Sommaire

ON A PROCEDE à une classification des principaux types des soupapes a vide employées pour isoler une partie d'un système sous vide d'un autre et on a décrit un grand nombre de modèles realisés durant les vingt, dernières années. On a considéré les exigences d'une soupape à vide et l'on a fait des critiques et des commentaries sure les modèles de soupapes déjà existants. On a décrit aussi bien les soupapes à vide (dèriveés de soupapes déjà existantes) utilisées généralement dans la pratique que les soupapes construits spécialement pour de très basses pressions. On trouvera de plus un rapport sur des études récents sur des soupapes pour des systèmes à vide très poussé, On a fait une brève discussion sur les possibilités de rèaliser dans le future de nouveaux modèles, principalement des soupapes pouvant être degazées par étuvage.

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REQUIREMENTS

TWENTY YEARS AGO, vacuum technique was almost exclusively an affair of small mechanical rotary pumps, mercury diffusion pumps, glass vessels and tubing, glass-to-metal seals and, inevitably, greased glass cones and stop-cocks. Developments since in vapour-stream pumps of the diffusion and ejector types having large speeds, and in the production of mechanical rotary pumps of high free air displacement, have led to new industrial methods employing vacua and new methods of designing and erecting vacuum equipment. As is well known, high pumping speed can only be obtained by the use of wide-bore, short, connecting tubing between the vapour pump and the vessel and also, though to a lesser extent, between the rotary pump and the vapour pump. Consequent on this necessity, there has been much progress in the design of mechanical valves with high conductance to gas flow to replace the glass stopcock and mercury cut-off which cannot be made with effective conductances of much greater than some 10 litres per second. Mechanical valves, largely fabricated from metal, have the additional advantages of robustness and freedom from the use of grease which, apart from its vapour pressure, is likely to release occluded air.

The requirements of an ideal vacuum valve to be employed for the isolation of one part of a vacuum system from another, *e.g.* as a cut-off between rotary pump and vapour pump or between vapour pump and vessel*, may be listed as follows:—

(i) They should not introduce significant impedance to the flow of gas through the system, *i.e.* they should not reduce seriously the through-

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* It is not proposed to deal here with needle valves and similar devices used for admitting a regulated small rate of flow of gas into a vessel, except insofar as some isolation valves may also be used for control purposes. Air-admittance valves are also excluded.

put of the system.

(ii) In most cases, they must be free from gas leaks when subjected to an external pressure of one atmosphere when a high vacuum exists internally.

(iii) Usually, mechanical strength is desirable.

(iv) Rapid operation and, in some applications, good regulation of gas flow is desirable.

(v) The action should be positive and not adversely influenced by gas pressure differentials across the valve.

(vi) The materials used should not evolve gases or vapours at temperatures below 30°C.

(vii) The construction materials used should be chemically inert to the gases and vapours handled. They should also be rust-proof.

(viii) The valves should be capable of being heated to permit outgassing.

In regard to these requirements, many designs have been published in the literature and commercial patterns are available which satisfy points (i) to (v) inclusive. The majority of valves do not evolve gases or vapours (see point vi) to any significant extent at room temperature where the ultimate pressure demand is not lower than 5×10^{-6} mm. Hg, yet further work on constructional materials is necessary to produce satisfactory valves where higher ultimate vacua are demanded. This is especially the case where it is required to maintain a high vacuum after the pumps have been cut off from the vessel by the valve in question. Thus, rubber and Neoprene gaskets are widely and successfully used in many valve designs, but both these materials are suspect from the point of view of outgassing at room temperature[1]. Requirement (vii) is satisfied by most types of valve for normal vacuum work where air is pumped, but further designs capable of withstanding corrosive vapours like bromine, ammonia, hydrochloric acid gas, etc. are needed, especially in chemical engineering practice. In this connection, it is unfortunate that several commercial patterns are fabricated from unplated mild steel and are, therefore, not rust-proof. It is only recently[2] that mechanical valves capable of being outgassed by baking at 300° to 400°C (see point viii) have been the subject of serious design work. Further work, especially towards making a commercial model with good gas conductance and able to withstand outgassing by heating, is urgently required.

In addition, mechanical valves for isolation purposes are sometimes required to be operated automatically at a predetermined pressure or time in the cycle of vacuum operations undertaken. Further, it is often desirable to be able to operate the vacuum valve from a position remote from the plant, or to operate simultaneously a number of valves on a complex vacuum system. Magnetic valves and also compressed air-operated valves are valuable for such applications.

DESIGN CHARACTERISTICS OF VACUUM VALVES

Classification by Structural Features

In attempting to classify the many patterns of vacuum valves designed for isolation purposes, it is best to consider two features essential to all models and detail the various methods of obtaining these features in practice. These two essentials are illustrated schematically in Fig. 1. They are :

(*a*) On closing the valve, a gas-tight seal must be effected where the valve plate or disc meets the the valve seat.

(*b*) The rod, shaft or other device used to transmit motion to the valve plate, situated inside the evacuated valve body, from the handle fitted to the outside, must pass through an air-tight seal provided in a convenient place in the valve wall or bonnet.

There are three methods in use for achieving (*a*). They may be classified as :

(1) The sealing materials are rubber or synthetic rubber, such as oil-resistant Neoprene. This is the most frequently adopted practice.

(2) The sealing material is silver chloride or a cement of similar properties.

(3) Two dissimilar metals, one hard and the other soft, are used as sealing materials pressed together under a high mechanically exerted pressure. Seals between two machined metal

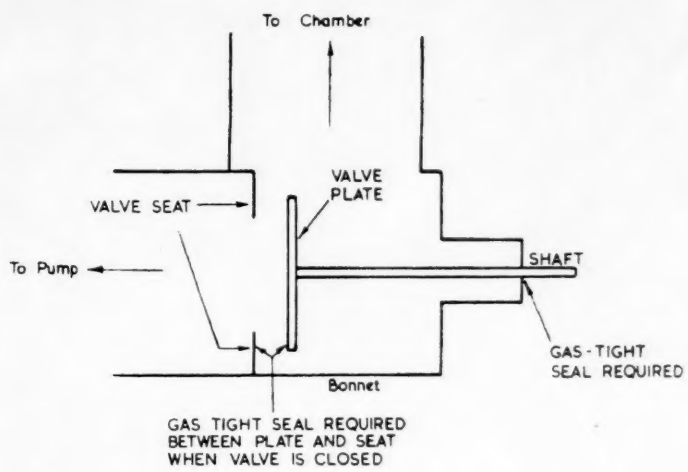

Fig. 1. Main features of vacuum valves for isolation purposes.

surfaces in contact under moderate mechanical pressure have also been employed, but these are liable to gas leakage.

There are two chief methods of satisfying (*b*). They are :

(4) The provision of packing, generally in the form of a rubber or Neoprene seal, as in a Wilson seal[3], or by using one or more ' O '-rings.

(5) The provision of Tombac or Sylphon bellows. Here the use of a flexible corrugated metal diaphragm can also be included.

Combinations of the three methods (*a*) with one or other of the methods (*b*) cover the principle characteristics of the valve types which have been developed. Differences remaining between the various patterns are concerned chiefly with methods of transmitting the mechanical motion, the materials used, and the application required. Multi-way valves, *e.g.*, a two-way valve as used between a rotary pump and a diffusion pump to provide pumping either *via* the diffusion pump or *via* a by-pass line straight to the vessel, are merely suitable mechanical arrangements which are extensions of the simple shut-off valve. Since such multi-way valves do not demand fundamentally different techniques they will not be specially considered here.

It is proposed to deal, in turn, with valve designs following the classification (*a*) 1, 2 and 3 and to discuss methods (*b*) as they arise.

(1) Valves with Rubber Seals

Two main methods of producing a satisfactory vacuum valve in this category have been adopted. Firstly, many workers have taken existing valves as used in general engineering liquid, gas or steam practice, and have modified them for vacuum work. In general, these modifications have been to dismantle the valve, clean it, machine certain surfaces to a higher degree of precision, and then introduce rubber discs, gaskets or ' O '-rings to ensure a vacuum-tight seal between the valve plate and seating when the valve is closed. The packing around the shaft from the valve handle in air to the plate or disc inside the valve is also removed and replaced by a rubber gland, a Wilson seal, an ' O '-ring, or by soldered-in Sylphon or Tombac bellows. Rubber gaskets are also usually greased with a low vapour pressure grease such as Apiezon. In some designs, certain parts of the valve

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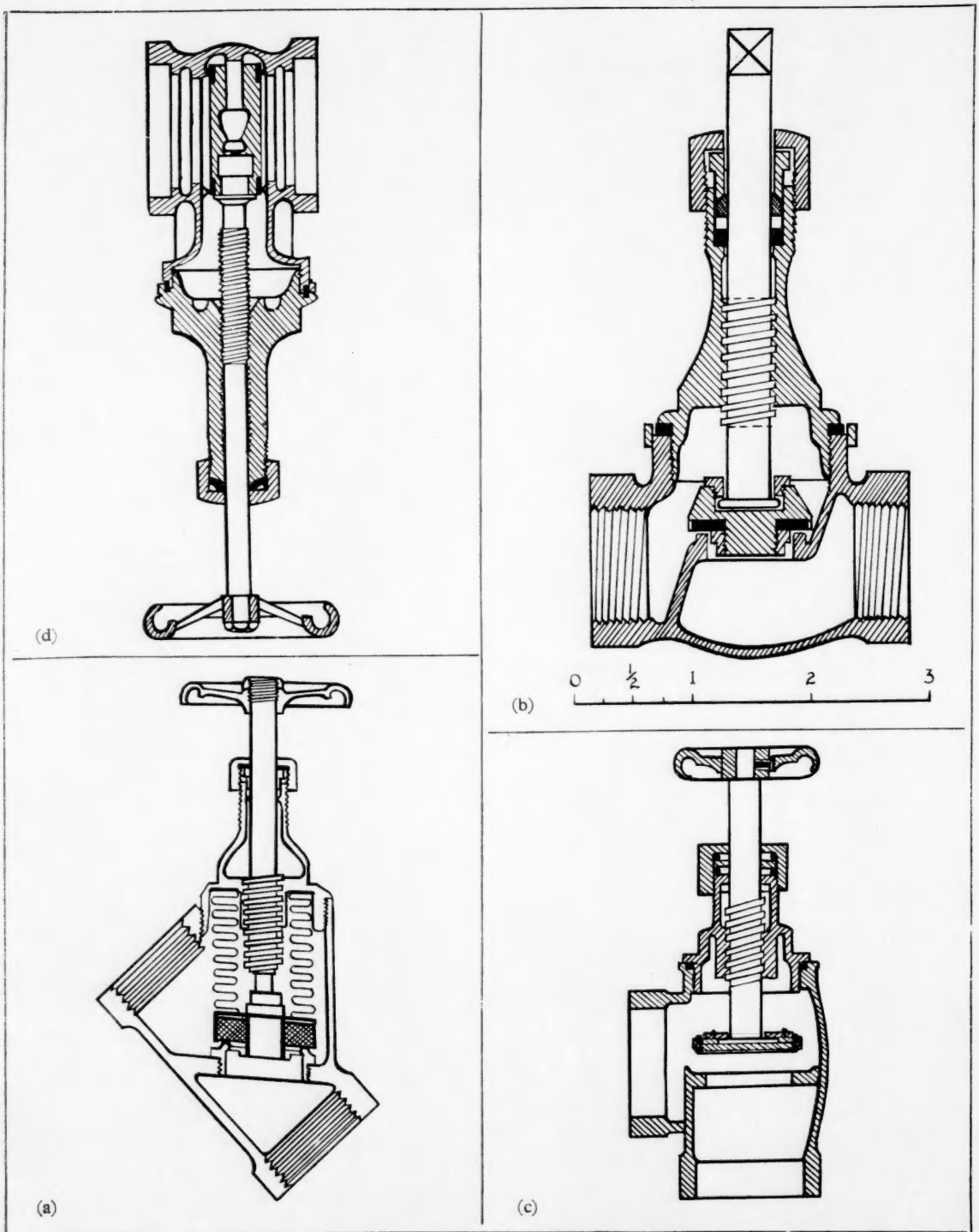

Fig. 2. Vacuum valves derived from general gas and steam valves.

have been electroplated to ensure corrosion-resistant surfaces. Generally speaking, valves with bore diameters from ½ inch to 2 inch. can be successfully, readily and economically manufactured in this way. They are extensively used in metal vacuum systems where pressures in the region of the valve not less than 1 micron Hg are demanded. Thus they are used where a mechanical rotary pump only is employed, or between the rotary fore-pump and a diffusion pump.

Typical of this practice are the valves designed by Dumond[4] and Rose[5], based on a Jenkins 1-inch Y steam-valve made of bronze (Fig. 2a); Moore[6], who preferred a Neoprene ring as packing since the use of flexible metal bellows restricts the distance through which the valve plate may be moved from its seating (Fig. 2b); Cowie and Green[7], who modified a Stockham 2-inch. angle steam valve (Fig. 2c); Kurie[8], who described a modification of a Crane No. 440 brass gate valve (Fig. 2d), alleged, because of its double disc, to have the virtues of high pumping speed and suitability as a vacuum lock enabling the insertion of items such as filaments or probes into a cyclotron, for example, without much disturbance of the main vacuum system.

Perhaps the Saunder's valve is the best known example in Britain of a valve developed for routine engineering use which can have its seating surfaces re-machined to make it suitable for vacuum purposes at pressures greater than 1 micron Hg (Fig. 3). This convenient and economical valve is available commercially in sizes from 0.5 inch to 2 inch.

The second method adopted has been to design valves specifically for vacuum systems. Usually, this applies to larger valves with bore diameters in the range from 2 inch. to 16 inch., and especially for operation in the fine-side region of the vacuum system where pressures down to 0.001 micron Hg may be encountered.

Such valves are most frequently encountered in the form of plate or disc valves for use between large port-diameter oil diffusion pumps and the vessel.

In this category, Krohn[9] describes a valve using a Neoprene gasket seal between the valve plate and the seat which has been made in diameters of 1 inch, 2 inch. and 6 inch. Metal bellows are used to provide a seal between the operating shaft and the bonnet of the valve. To avoid undue length of travel of these bellows and so prolong their operational life, and yet permit adequate motion of the valve plate, a rack and pinion mechanism was used on the vacuum side. A minor feature of this valve worth noting is that all close-fitting parts such as screws, etc. had small pump-

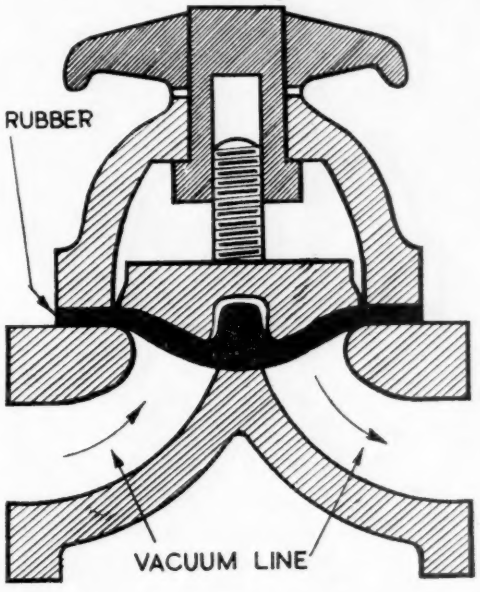

Fig. 3. Typical weir-type diaphragm valve.

out grooves to permit rapid evacuation of air pockets. Grivet and Blattmann[10] also give a design of valve in which the bellows extension is limited to 15% but where a much greater translatory motion is imparted to the valve plate by the use of a coupling mechanism consisting of jointed metal bars forming a set of articulated parallelograms, reminiscent of trellis work. D'Eustachio[11] has designed a valve with a square plate, the motion of which was guided by four rods

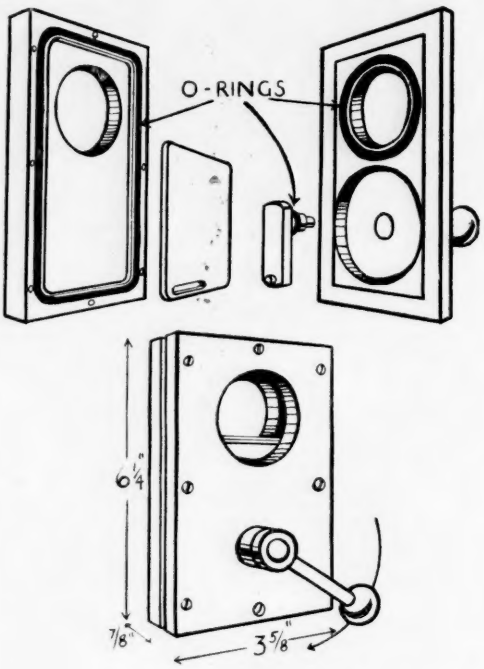

Fig. 4. A sliding flat-vane valve.

and which was cam-driven. Helical springs on the rods pushed against this plate and ensured that it followed the cam motion. The rubber gasket seal used was in a suitably shaped groove in the valve seat. This worker recommends heating such gaskets to 70 to 80°C in vacuum before use—a useful precaution to reduce subsequent outgassing. D'Eustachio also made a similar valve with fused silver chloride in place of the rubber gasket[25]. The valve seat was then made from stainless steel and the silver chloride fused into the groove by heating rapidly to 500°C and then cooling slowly. A polished, machined circular ridge on the valve plate, which corresponded with the channel in the seat, was rotated with considerable pressure against the silver chloride to polish it prior to the assembly of the valve. A force of 20 to 25 lb. cm. length of the silver chloride seal was recommended. The cam shaft seal used consisted of coarse steel wool sandwiched between two pads of long-fibre asbestos. The steel wool was saturated with diffusion pump oil. Using a silver chloride seal and silicone pump fluid in the shaft seal, with cooling water around the valve at 5°C, D'Eustachio claimed that a pressure of 10^{-7} mm. Hg could be attained in a system in which this valve was incorporated.

Jennings[12] describes a valve which has a large straight-through passage when in the open position. This is particularly necessary for use in Van Der Graaff generators and cyclotrons of the external ion beam type so that the ion beam, entering through the open valve, cannot ' splash ' on the valve parts.

This valve can also be closed rapidly in case of a breakdown—a particularly likely possibility in generators for accelerating fundamental particles to high energies. A conventional ' O '-ring is used in a channel in the valve plate to form a seal against the seat in the closed position. The shaft into the valve bonnet was rendered vacuum-tight by a Wilson seal. The mechanical force necessary to press the valve plate against the seat is provided by a lever structure consisting of three bars, one, a radius bar to hold the valve centrally over the seat, and the other two to form a toggle joint connecting to an external valve handle in atmospheric air.

Lockenvitz, Hughes, Lipson and Olewin[13] give a design for a high vacuum valve for use on a mass spectrograph for the analysis of solid samples. Sylphon bellows are used to render the valve shaft air-tight. A feature of this valve was that two valve plates, each furnished with rubber gaskets, were used so that, in the open position, the air inlet, the exhaust hole to the auxiliary pump of the mass spectrograph, and the Sylphon bellows were sealed from the main vacuum system.

King [14] describes a valve in which the gate comprises two rigid, flat surfaces, both of which are inserted in the main 8-inch. diameter vacuum pipe line. These plates are both furnished with rubber gaskets in grooves. To permit ready insertion and withdrawal of this pair of plates from the side port into the pipe line, the plates are normally drawn together by springs. On insertion, the plates are then forced apart against the valve seats by a cam fixed on the

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operating rod. A Wilson seal is used to seal the driving shaft from the bonnet of the valve.

Further designs are considered by McIntosh and Coltman[15] and by Asao and Muramatsu[16], who used rubber gaskets for sealing all the metal parts of the valve bonnet so as to avoid welding or soldering.

A novel design, introducing considerable simplification in the operating mechanism, and providing a valve of low impedance to gas flow, is due to Wahl, Forbes, Nyer and Little[17]. This may be classified as a sliding flat vane type (Fig. 4).

In essence, supposing the straight cylindrical pipe line between the diffusion pump and the vessel is vertical, then the flat valve plate is turned in a horizontal plane into the side of this pipe to form a simple cut-off. To ensure a satisfactory seal, the valve plate, in fact, slides between two fixed horizontal metal plates set at about $\frac{3}{8}$ inch apart. These fixed plates are furnished with central circular apertures, coinciding with the pipe bore, and are welded around the pipe line. Neoprene 'O'-rings are set in suitably cut channels in the inner flat surfaces of these plates, these rings being concentric and outside the bore. The moving plate or vane hence slides between the two 'O'-rings in the act of closing or opening the valve. To reduce friction, these rings are lubricated with silicone grease. It is reported that this arrangement makes a satisfactory seal. Apart from some doubt in this respect, the arrangement is attractive mechanically since it is apparent that the seal between the shaft and valve bonnet, essential in previous patterns, is avoided. A disadvantage is that there is a tendency for the 'O'-rings to be pushed out of their channels by the sliding vane. To reduce this possibility, the 'O'-rings employed are one size smaller than those normally recommended for the diameter of

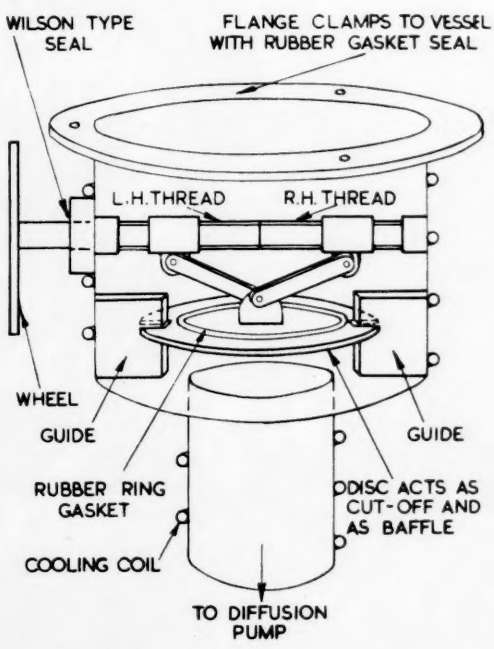
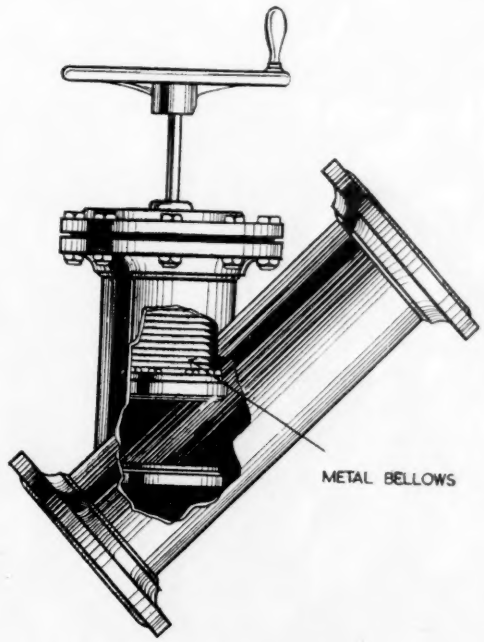


Fig. 5 (a). Typical water-cooled baffle valve.



(b) Typical bellows-sealed 4-valve.

channel used, and hence have to be stretched in insertion. Bondelid[18] reduces the possibility of ' O '-ring displacement in a modified valve, in which inset circular metal pins are used as spacers to enable the vane to be moved free of the ' O '-rings until the final $\frac{1}{2}$ inch of its motion.

Smith and Seagondollar[19] also describe a sliding vane valve where one plate slides over another which contains an inset ' O '-ring. To provide the force

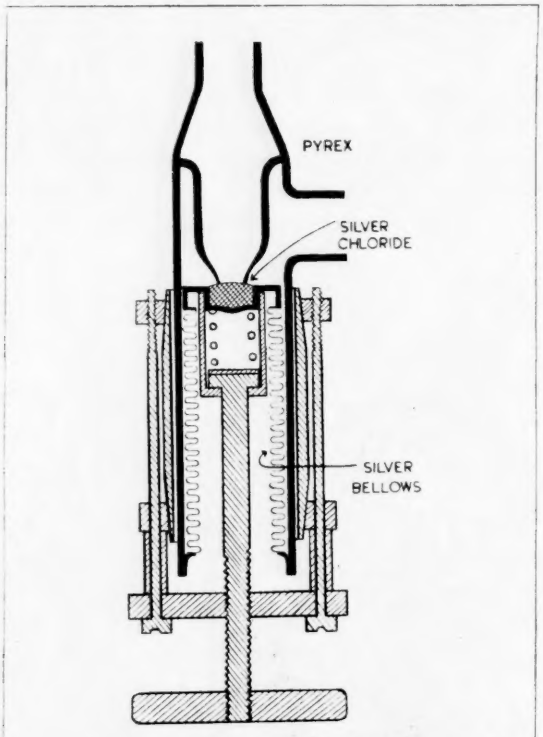


Fig. 6. A greaseless and chemically inert valve (Rampsberger).

necessary to overcome the resistance to motion due to friction, a worm-gear drive was employed. Valves of $\frac{1}{8}$ inch and 1 inch bore were made in this way.

Garrod[20] employed Neoprene washer sealing and a Wilson type shaft seal in a multiple high-vacuum valve designed to open or close simultaneously or independently four 1-inch diameter pipe lines. A feature of the mild steel cylindrical pot used as the valve bonnet was that the permanent vacuum-tight seals necessary were made between the screwed-together parts with the help of a loop of 20-ampere fuse wire. Two other designs have also been published by this worker[21]. Riddiford and Lilley[22] discuss the use of ' O '-rings and Wilson seals in the design of high vacuum valves with air-locks, to enable equipment to be passed by means of a shaft from the air-lock to the main vessel. These writers also give designs for a two-way double gate valve and a straight-through plate valve. Shatford[23] describes a simple isolation valve and a two-way valve in which a polished cylinder of metal is pushed between ' O '-rings; the shaft sealing is also done with ' O '-rings. Such valves are simple to construct and useful in bore sizes from $\frac{1}{4}$ inch to 1 inch. The two-way model is particularly useful between the rotary and diffusion pumps when a by-pass line is provided. Morton and Jeffery[24] gave a design of a sliding gate-type of valve which offers very little obstruction to the flow of gas when the valve is open.

The commercial designs of large diameter valves for isolation purposes between the diffusion pump and the vessel, generally use circular valve plates furnished with ' O '-rings in a suitable groove. Typical of these are the designs of Edwards High Vacuum Ltd., who employ a Wilson type seal for the shaft (Fig. 5a), and those of the National Research Corporation (U.S.A.) where bellows shaft sealing is used (Fig. 5b).

(2) Valves with Silver Chloride Seals

Rampsberger[25] described a type of valve which, so far as is known, has been little used but which might well find applications in research practice, where pressures at less than 10^{-6} mm. Hg are required in a glass system. A seal is effected between fused silver chloride and the orifice at the mouth of a shaped Pyrex glass tube when the valve is closed (Fig. 6). Silver bellows are fused to a glass tube at their lower end whilst, at their upper end, a cup-shaped depression contains the silver chloride. The valve is operated by the screw attachment shown, the working of which is obvious from the diagram.

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(3) Valves with Metal-to-Metal Seals

It is attractive to dispense with grease or rubber as a sealing agent because both these substances give off gases and vapours at low pressures. The possibility exists of forming a valve by simply pressing one machined metal surface against another. However, it is difficult, if not impossible, to ensure so good a fit in practice as to forbid gas leakage unless the surfaces are under a powerful compressive force. Nevertheless, attempts at making valves without the use of such high forces have been made, and, provided that a small gas leak through the closed valve is permissible, they have found useful applications.

Lang[26] describes a valve (Fig. 7) that does not employ mercury, grease or rubber, for use on a vacuum spectrograph. Since cast brass is porous, this valve was fabricated from drawn brass. The essential valve seat consisted simply of a raised rim of soft brass about $\frac{1}{8}$ inch wide and $\frac{1}{16}$ inch high, whilst the valve plate was a piece of soft brass having a copper plate inset in its lower face; this copper fitted over the seat when the valve was closed. The surfaces involved were not ground, but finished smooth on the lathe. A ball and socket joint was used to attach the spindle to the cover plate to prevent the copper plate from damage on the seat on rotation of the spindle when the valve was tightly closed. To ensure freedom from leakage between the movable spindle and the bonnet of the valve, a fibre washer and two packing glands were used, and the space between the bonnet and the spindle was filled with Apiezon grease introduced under pressure through a nipple using a grease-gun. Thus, whilst the vacuum vessel was not exposed to the grease and packing gland when the valve was closed, it was, nevertheless, exposed by an indirect path of high impedance when the valve was open and the vessel was undergoing pumping.

Esterman and Foner[27] describe a valve for sealing off a port diameter of $3\frac{1}{2}$ inch. which does not use rubber, provided a gas flow of a few c.c./sec. is permissible when the valve is closed, with the advantage that a flow of a hundred litres per second or more is obtainable when open (Fig. 8).

The valve plate consists of a circular brass disc with

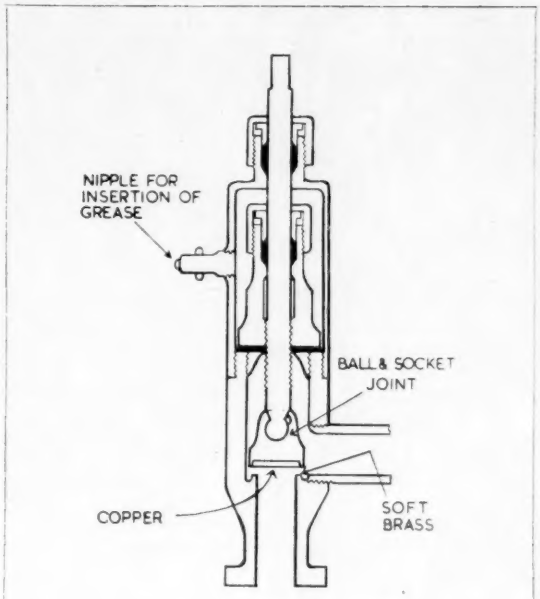

Fig. 7. Vacuum valve (Lang).

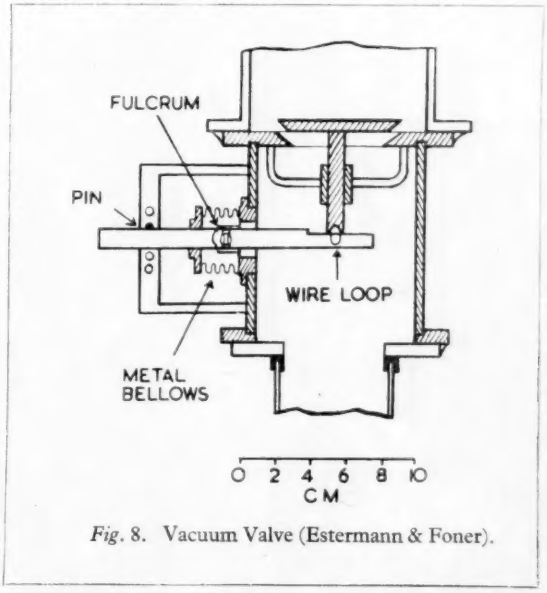

Fig. 8. Vacuum Valve (Estermann & Foner).

a ground conical edge which fits, when the valve is closed, into the correspondingly ground edge of a circular aperture in a brass plate as the valve seat. Monel metal or stainless steel could be used in place of brass if increased inertness to chemical attack were necessary. The valve disc is movable relatively to the valve seat by a lever through Tombac or Sylphon bellows. To minimise stresses produced in these bellows during the motion of the lever, the lever fulcrum required was located inside the bellows. No attempt to lap the valve plate and seat was made. It was felt that, if such proper grinding were employed, then leakage through the valve when closed could be reduced to less than 1 c.c./sec. at 1 micron Hg.

The most notable of the recent work on vacuum valves, which dispenses with the use of rubber, grease or mercury, is that of Alpert[2]. This valve has the great advantage that it can be degassed by baking at 400°C. As a cut-off valve for use in ultra-high vacuum work, *i.e.* at pressures less than 10^{-6} mm. Hg, it was an outstanding innovation. The Alpert valve has only been made with an orifice diameter of $\frac{1}{4}$ inch, but larger sizes have been produced in a modified design due to Brown and Coyle[28].

SPECIAL PURPOSE VACUUM VALVES

Valves for Ultra-High Vacua

The Alpert valve (Fig. 9) consists of two sections : The valve assembly (Fig. 9a) and the driver mechanism (Fig. 9b), which opens and closes the valve. During a degassing bake at some 400°C, this driver mechanism is taken off and the valve retained in its open position by a U-shaped stainless steel clamp, called a ' bake-out ' clamp.

The valve seat consists of a copper cup of $1\frac{3}{4}$ inch. diameter with two orifices, each $\frac{1}{4}$ inch diameter (Fig. 9d). The valve disc or plate is a Kovar nose-piece which has a highly polished 45° conical surface which forms its own seat in the copper cup when the valve is closed for the first time. To provide a seal between this movable Kovar nose-piece and the valve bonnet, a flexible Kovar diaphragm, which permits a translatory movement of only 0.1 inch, is used. To ensure that the valve is extremely clean and free of surface oxides, all the joints necessary are brazed in a hydrogen furnace using silver-copper solder without flux.

The driver mechanism is screwed into position only when the valve is cold. It consists of a differential screw mechanism which moves the Kovar nose about 0.01 inch per revolution, the total travel being 0.1 inch. The driver mechanism comprises four parts : (i) an outside screw and housing of stainless steel, (ii) a silicon-bronze differential screw with a thread pitch of 20 per inch outside and 26 per inch inside, (iii) the stainless steel drive screw which is fastened to the valve nose, (iv) a stainless steel backing plate, securely and rigidly bolted to a bracket on the bench on which the vacuum system is mounted. The differential screw has a $\frac{1}{2}$-inch square head which is rotated by a torque wrench. The enormous mechanical advantage afforded enables a force of 5 to 10 tons weight to be exerted on the Kovar nose, forcing it against the $\frac{1}{4}$-inch orifice in the copper cup to give a vacuum seal with an effective conductance to nitrogen of only 10^{-11} litre per second. Even after a year's use, the conductance of this valve when closed increased to only 10^{-10} litres per second, the deterioration being due to slight oxidation of the smooth metallic surfaces and to the collection of minute foreign particles on the valve seat. With this valve, and using a vacuum system in glass which was sealed to the valves by glass-metal unions, Alpert obtained vacua of 10^{-10} mm. Hg, an ionisation gauge being used as a pump in the final stages of evacuation.

Brown and Coyle[28] give a design of valve for ultra-high vacuum work which they claim is more simply constructed than Alpert's version, from which it is developed, and yet it enables pressures down to 10^{-9} mm. Hg to be attained. The valve closure is made by a metal nose of Monel instead of Kovar pushing into a soft copper seat, and a larger orifice diameter than that in Alpert's pattern is achieved. The body and nut are of stainless steel, and the diaphragm of Monel metal is 0.004 inch thick. To facilitate soldering in a hydrogen furnace, the stainless steel body is nickel-plated to a thickness of 0.002 inch, and a carefully arranged soldering schedule is followed in the assembly of the valve. An interesting observation by these workers is that they used molybdenum disulphide as

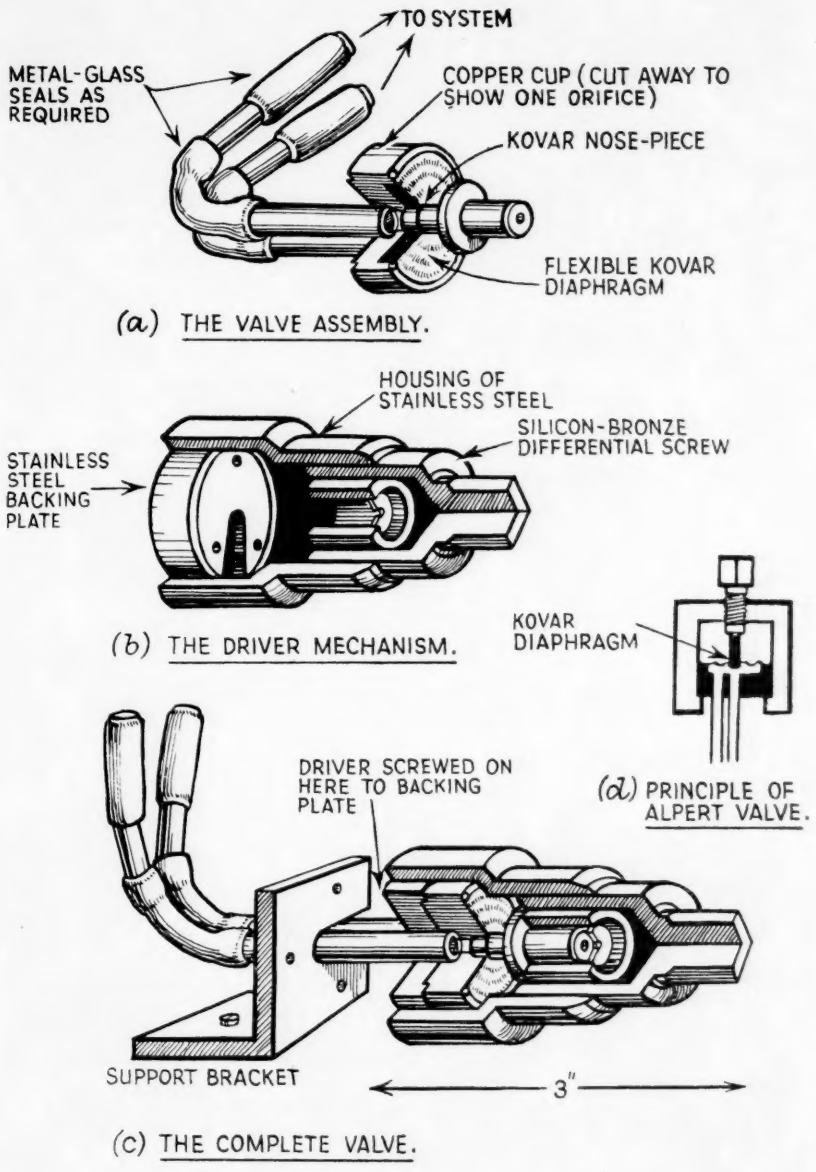

Fig. 9. Valve for ultra-high vacuum work (Alpert).

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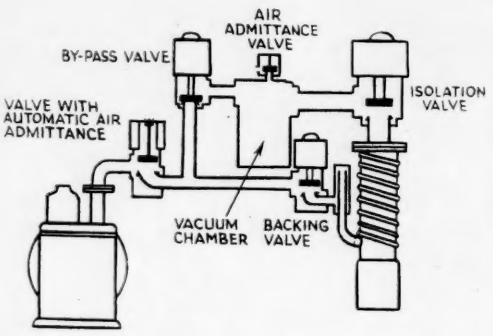

Fig. 11. Diagrammatic layout showing an application of magnetic valves in an automatic system.

a lubricant in this valve, and that this substance withstood successfully the vacuum baking at 400°C.

Automatic Vacuum Valves

To achieve an automatic or semi-automatic operation of the valves of a vacuum system, magnetically or compressed-air operated valves are used (Fig. 10). Magnetic valves are valuable in that they can be opened or closed at a predetermined pressure in the vacuum cycle. Thus it is a straightforward matter to arrange that the pressure-dependent e.m.f. available from a gauge of the Pirani or thermocouple-type be amplified and then made to operate a relay which energises or de-energises the solenoid of a magnetic valve of the type shown in Fig. 10b. Magnetic valves can also be usefully employed to shut off the pipe-line in the event of the failure of the rotary pump motor. Fig. 11 indicates possible positions of magnetic valves in a vacuum system. Both compressed air and magnetic valves can be applied to a complex vacuum plant where a number of valves must be operated simultaneously or at specified times in the cycle of operations.

Green[29] has devised a convenient automatic valve which protects a vacuum system against failure of the mechanical rotary pump due to either breakdown of the mains supply to the motor drive or due to break-

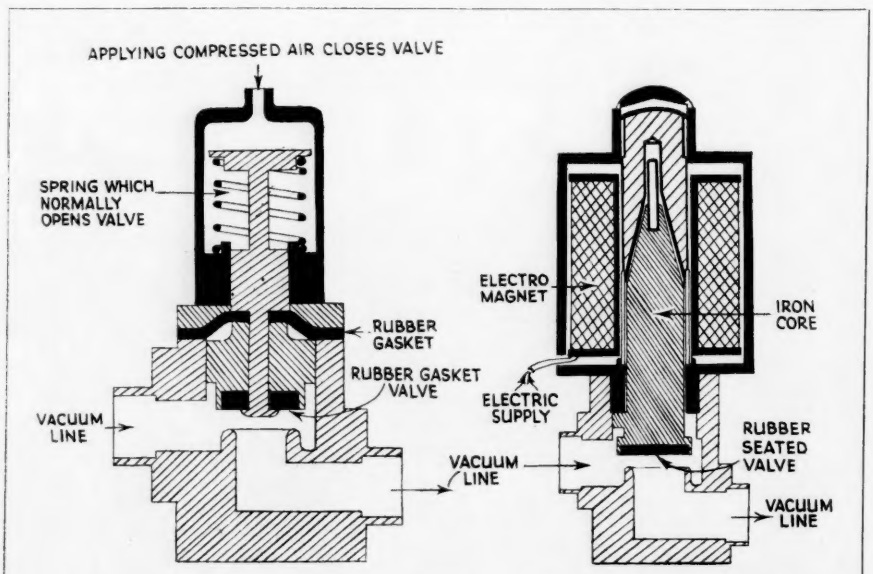

Fig. 10. Representative designs of compressed air and magnetically operated valves.

age of the belt drive. By the use of a single unit comprising a magnetically controlled leak valve in conjunction with a rubber-faced valve operated by the compression of Tombac bellows under air pressure, the protection afforded is twofold : the backing line is closed and air is admitted to the rotary pump to prevent oil from being driven into the backing line.

SOME SUGGESTIONS FOR FURTHER DESIGNS

There are three materials which might well be tried in the design of vacuum valves. Firstly, the possibility exists of using Teflon or copper-Fluon in place of rubber or Neoprene, especially to seal the valve operating shaft against the valve bonnet. Thus Teflon—the trade name for the plastic tetrafluorethylene resin—has good electrical, thermal and chemical resistant properties and is remarkably friction-free. It can be used over the temperature range from – 320°F to 550°F and has a vapour pressure[30] of 10^{-6} mm. Hg at 25°C. Rueger[31] discusses the use of a Teflon insert over the tip of the needle of a needle valve. This, it is reputed, ensures that the needle valve remains leak-free when closed after prolonged use. Secondly, silicone rubbers[32], which withstand baking to 250°C, might be used in place of Neoprene and permit a valve design which withstands de-gassing bakes. The chief difficulty here, however, is that silicone rubbers suffer permanent deformation even at moderate temperatures. Thirdly, the common metal tin, which has unique vapour pressure properties, might well be used for making a seal. The possibilities of tin rest upon the fact that, whereas its melting point is only 232°C, yet its boiling point is 2,270°C. Moreover, the vapour pressure of tin[33] is only 10^{-5} mm. Hg at 823°C. Thus tin could be used to make a seal which could be made or broken by raising the temperature to 232°C by electric heating, and by moving the valve plate towards, or withdrawing it from, the seat, both plate and seat being of a suitable shape and position.

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TRAINING REPORTS

THIN FILM OPTICS AT THE IMPERIAL COLLEGE OF SCIENCE AND TECHNOLOGY

THE SUBJECT OF Thin Films Optics has been a part of the one-year postgraduate course in Technical Optics given in the Technical Optics Section of the College since the session 1951-52. About six lectures are given on the theory of multilayers and their applications to optical instrumentation and experiments are arranged in which the students make interference filters, etc.

This session (1954-55) it was decided to arrange a public lecture course and to enlarge the scope considerably. There were eight lectures and also a practical course was offered. The total attendance at the lectures was 39; of these 19 were Imperial College students (undergraduate and post-graduate), 14 were from industrial establishments and 6 from other colleges in the London area. Four also took the practical course. Summaries of the ground covered are given below.

Lecture Course

(1) Plane electromagnetic waves in a stratified medium. Derivation of the basic recurrence relations between the complex amplitudes of the electric vectors in successive layers. Reflection and transmission factors in terms of the complex amplitudes.

(2) Various forms of the recurrence relations, involving (*a*) complex amplitudes of electric vectors of waves travelling in positive and negative directions, (*b*) complex amplitudes of total tangential components of electric and magnetic vectors (*c*) amplitude reflection and transmission factors and (*d*) admittance.

(3) Matrix expression of the recurrence relations (*b*) above. Numerical computation using matrices, including checks. Use of the matrix method for determining the effect of a change in one layer. Graphical computation using the admittance formulae with Smith charts.

(4) Multilayers with continuously varying refractive index. Anti-reflection coatings with one and two layers. A numerical example illustrating the design of a two layer anti-reflection coat by an approximate graphical method and verification by matrix calculation. Simulation of a coat of arbitrary refractive index by a symmetrical triple layer using given indices.

(5) High-reflecting all-dielectric layers. The matrix for a periodic multilayer of n periods. Reflecting and transmitting zones. Effect of varying the thickness ratio in the fundamental period. Dichroic beam-splitters. Reduction of subsidiary reflection bands in the transmitting zones by altering thickness ratio and by trimmer layers.

(6) Narrow transmission band interference filters. Metal-dielectric type : theory of band width, maximum transmission and polarisation splitting on oblique incidence. All-dielectric type : effect of phase change at high reflecting layer on band width.

(7) Hybrid types of interference filter. Reflection interference filter, frustrated total reflection filter. Multilayers as polarising devices.

(8) Methods of measurement of refractive index and control of film thickness. Photometric and polarimetric methods for use in vacuum chamber. Measuring phase retardation and thickness in air by Zernike and Tolansky methods. Polarisation and photometric methods in air. A list of available materials with refractive indices.

In the lecture course the derivation of the theory was separated from the practical applications in such a way that those who wished could use the computational techniques without following the theory in detail; this was possible because the practical details of matrix computation are essentially very simple.

Practical Course

The following experiments were arranged :

- Blooming and aluminising.
- Metal-dielectric interference filters.
- Enhancing reflectivity of a metal layer with dielectric coatings.
- Evaporating quartz as a protective layer.
- High-reflecting all-dielectric coatings.
- Measurement of film thickness by multiple-beam fringes.
- In addition, exercises on matrix computing were carried out.

The lectures were given by Dr. W. Weinstein and the practical course was supervised by Messrs. J. A. Dobrowolski, A. Musset and Dr. Weinstein. At the end of the course about half of those attending visited the laboratories of a leading vacuum plant manufacturer.

W. WEINSTEIN.

LETTERS TO THE EDITOR

A Stainless Steel Trap for a Metal Vacuum System

Sommaire

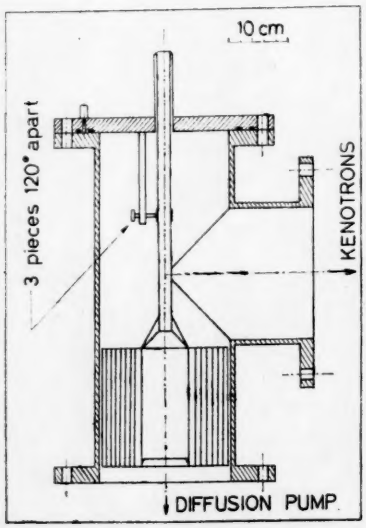
APPLIQUANT UN PROCEDE qui s'est montré efficace dans les pièges en verre, on a placé une spirale de tôle ondulée d'acier inoxydable dans un piège métallique, ce qui permet de réduire la consommation d'air liquide.

WE HAVE CONSTRUCTED a trap of the type developed by D. Alpert[1] and inserted it between the diffusion pump and the kenotron column which forms part of the Cockroft-Walton accelerator in our laboratory. Fig. 2 gives the plan view of the trap and Fig. 1 its position in the vacuum system.

The trap is made in stainless steel because we intend to exchange the oil diffusion pump, at present in use, against a mercury diffusion pump.

The sheet placed in the trap is 0.1 mm. thick and 100 mm. wide and has been corrugated in an apparatus of the type described by Alpert[1]. We built one of these units ourselves for the purpose.

Fig. 1



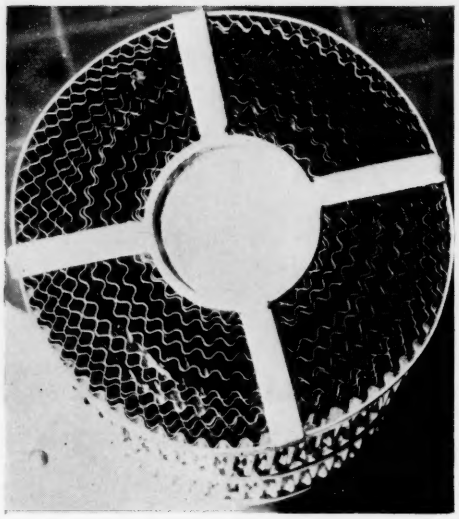


Fig. 2

It consists of 2 splined rollers (30 splines p. roller, modulus=2.5). The distance between the (horizontal) axes of the rollers can be varied according to the thickness of the sheet to be corrugated. The depth of the corrugation is 5.4 mm. and the distance between the splines is 7.8 mm. The apparatus takes sheets of up to 300 mm. width.

With the trap previously used the consumption of liquid air was about 2 litres per hour. After fitting the new trap we found that the consumption was reduced to about 0.3 litres per hour.

The sheet is inserted into the trap in the manner shown in Fig. 1, *i.e.*, the corrugations run parallel to the axis of the trap. But this method of assembling the sheet has the drawback that in some regions two adjacent sectors tend to 'fit snugly', as shown in Fig. 2, in the area near the axis. We believe we can overcome this and improve the design of the trap by inserting one flat sheet between two corrugated sectors. Fig. 3 depicts a prototype of the new design. It shows a much more regular spacing of the corrugated sheet.

Nuclear Physics Centre,
Royal Military School,
Brussels, Belgium.

E. THOMAS,
J. DESTAPPES
J. DUPONT.

10th March, 1955.

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Fig. 3. (Stainless Steel Trap)

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Polythene Windows for Vacuum Systems

Sommaire

UNE MÉTHODE EST décrite brievement pour la fabrication des fenêtres à partir de ruban de polythène en tubes qui peuvent servir comme fenêtres de tubes à rayons-X et donnent une distribution très large d'irradiation de rayons X qui est désirable dans certaines recherches spéciales.

A HOLE OF THE required size is made in the glass tube by grinding with a suitable grinding paste. The sharp edges are then removed with a fine carborundum stone. An ' O '-ring is selected of such a size that it will just rest on the circumference of the hole. The cross-sectional diameter of the ' O '-ring should be approximately 0.140 inch. A track $\frac{1}{8}$ inch wide around the hole is then treated with adhesive S 21, (a specially prepared rubber adhesive). This is done by lightly painting with S 21, allowing to dry 3-4 minutes and then painting with S 21 again. The ' O '-ring is pressed into position and a slight pressure is maintained for approximately 10 minutes. The ' O '-ring will now take the shape of the contour of the glass tube.

Windows of 0.010 inch polythene tape can be prepared by wrapping the polythene tape around the tube over the sealing ring and spot welding the two ends together on the opposite side of the tube. The spot welding can be done quite well with a soldering iron.

Test Procedure. Both ends of a tube similar to the one described in Fig. 2 were sealed and pumped through a smaller tube situated on the side of the main tube (see Fig. 2). A vacuum of 5×10^{-6} mm. Hg was readily attained. The polythene sheet can be easily removed and refitted or replaced by a new sheet and the tube can be rapidly evacuated after the operation.

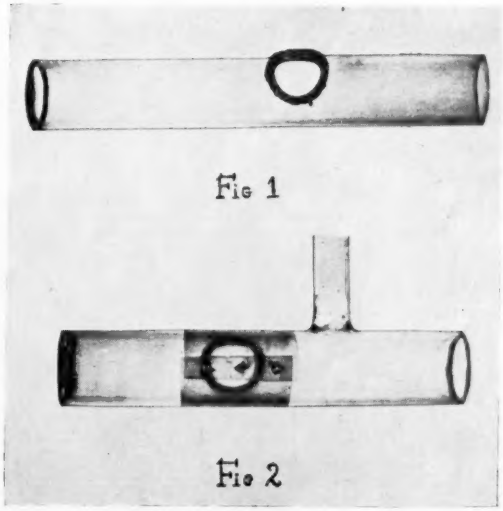
Fig. 1 shows a 35 mm. diameter lead glass tube with a hole of 1 inch diameter and with a rubber sealing ring fixed to the circumference of the hole with S 21 adhesive.

Fig. 2 shows the same tube as Fig. 1 but in this case the 0.01 inch polythene tape has been wrapped over the ' O '-ring and around the tube; both ends have been spot welded together on the opposite side. The tube has been fitted with a smaller diameter side tubulation for evacuation purpose.

Application. This method has proved very useful as a method of constructing X-ray tube windows in cases where the window is required to be as near as possible to the source from which the X-rays are being emitted.

As soon as the tube has been evacuated the polythene is drawn inwards and a window of the size described above (using 0.01 inch tape) would move inwards by as much as 0.15 inch at the central portion of its effective area. In the particular case in which

Polythene Window



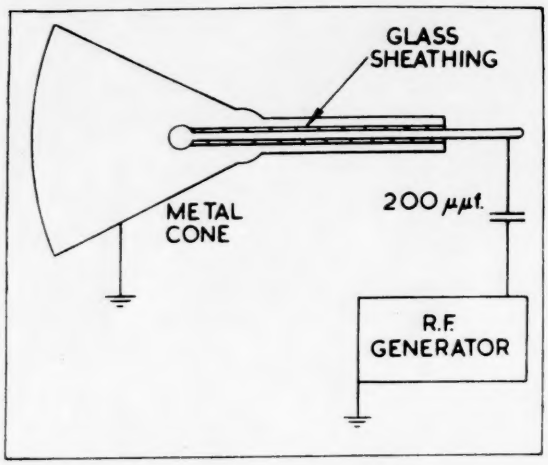


Fig. 1. (Screen Inspection Method)

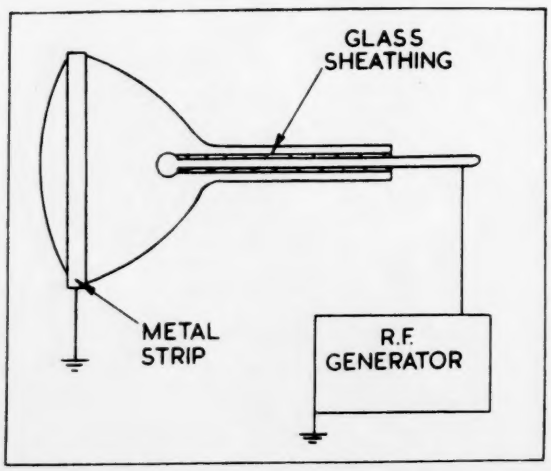
this window is used on a demountable X-ray tube this movement inwards leads to the following advantages :—

(1) The surface of the window comes slightly nearer to the target.
(2) The angle of distribution of the X-ray radiation is wider.
(3) The junction between the polythene and the 'O'-ring is perfectly vacuum tight.

Operational Test. An X-ray tube was fitted with this type of window and was operated with 13 kV on the anode and a target current of 1 mA for 10 seconds. This was repeated 60 times at ½ minute intervals. The target voltage was left on all the time but the filament was turned off completely during each rest period.

The 0.010 inch polythene window was quite satisfactory after this test which involves, of course, only restricted irradiation by the X-ray beam. If the tube was operated continuously at 5 mA the window would fail after about 60 minutes operation. On the other hand, if the tube was allowed to remain under vacuum but without production of X-rays, the window would last almost indefinitely. The polythene window is therefore not at all durable as a window for a demountable X-ray tube, but it has a long enough life to enable certain experiments to be carried out, in which it is necessary to use a powerful beam of very low voltage X-rays and of very wide angle of distribution for certain special investigations.

Fig. 2. (Screen Inspection Method)



J. HODKINSON.

Physical Laboratory,
Ferranti Ltd.,
Moston, Manchester 10, England.

2nd March, 1955.

An Inspection Method for Luminescent Screens

Sommaire

THE DECHARGE de gaz à basse pression sous haute tension dans un tube cathodique excite l'écran luminescent. Ce phénomène a été etudié pour son emploi dans une méthode d'inspection d'écran.

THE DETAILED NATURE of screen luminescence excited by one agent can be very different from that excited by another. The exact nature of luminescence depends on :—The energy distribution, or energy spread of the exciting agent; the kind of exciting agent, whether photons, electrons, or other particles; and also on the direction from which the exciting agent is applied.

In this sense, since the screen, in normal use, will be bombarded from the rear with electrons having only a narrow energy spread, the ideal would be to excite the screen in precisely this manner. Other tests, such as ultra-violet light directed onto the front of the screen, may or may not show imperfections, which, in turn, may or may not show under operating conditions.

Ultra-violet light frontal illumination has in fact

been found to give results, very different from electron bombardment of the rear. Its unreliability operates in both directions. It shows up imperfections which in a completed television tube would not be discernible, and also it fails to show certain other flaws, which are clearly visible under electron bombardment.

A method, employing a low pressure high voltage gas discharge, will be described below which gives satisfactory results.

Details of the Inspection Procedure

A very good approximation to electron bombardment of the rear of the screen is produced by a low pressure gas discharge where the screen forms one electrode. An aluminised screen, or a screen with a similar conductive coating, can be used as an electrode, but it is difficult to obtain uniform illumination of an insulated screen in a d.c. discharge. With a radio frequency gas discharge however, a screen which is insulated may act as one electrode by capacitative coupling.

The potential difference across a gas discharge may be varied at will by controlling the gas pressure, and hence the mean energy of the ions bombarding the screen may be selected as desired.

The requirements for a practical inspection unit are as follows :—A source of radio frequency power capable of an output e.m.f. of from 5 to 9 thousand volts, and a vacuum system capable of creating and maintaining a pressure of from 5×10^{-4} mm. Hg to 5×10^{-3} mm. Hg.

The arrangement we have tried successfully is as follows :—

Electrical Circuit. The anode of the cathode ray tube is earthed directly (Fig. 1), or if this is not possible, its capacity to earth is increased by placing an earthed metal strip, some 2 inch. wide, around the periphery of the screen end of the bulb (Fig. 2). A probe electrode carrying a $\frac{3}{4}$-inch diameter polished aluminium sphere at its extremity is inserted up the neck of the bulb so as to place the sphere about 7 inch. from the screen of a 12 inch. bulb, or at a geometrically similar position in other sizes. The metal rod supporting this sphere must be insulated (for example with glass tubing) over the portion of its length which is within the vacuum except where it actually joins the sphere (Fig. 1 and 2).

This probe electrode, where it projects outside the vacuum, is connected to a source of high voltage R.F. power. In our experiments a direct connection to the tank coil of a 1 kW dielectric heating set was found to be ideal. If the anode of the bulb has been directly earthed, a condenser, of some 200 pf and 10 kV working, must be placed in series with the R.F. supply lead, to limit the current (Fig. 1).

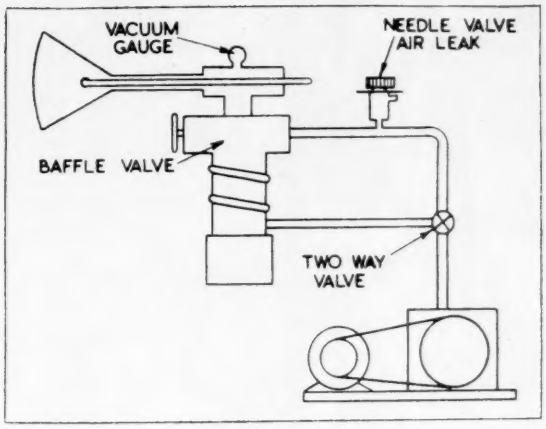
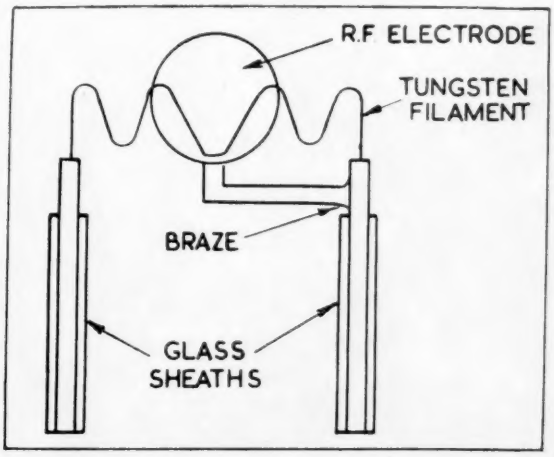


Fig. 3. (Screen Inspection Method)

Vacuum System. Heating, accompanied by a rapid evolution of gas, occurs when the gas discharge is operating. To maintain a constant pressure, a continuous flow method, having a rapid pump and a rapid air leak, must be used. If the quantity of air leaking into the system is great enough, it will reduce the pressure fluctuations, caused by varying rates of outgassing, to negligible proportions.

A diagrammatic representation of the vacuum system we have used is shown in Fig. 3. An oil

Fig. 4. (Screen Inspection Method)



diffusion pump, in combination with a baffle valve, giving a pumping speed of 50 litres per second, was in excess of the minimum speed required. A needle valve controlled the rate of air leak, and a single-stage rotary oil pump with a speed of 50 l./min. provided the fore vacuum. The latter was not quite fast enough to utilise the full speed of the diffusion pump, and in our experiments the baffle valve was partially closed to reduce the throughput of air to an amount, the backing pump could handle.

It was found unnecessary to include a moisture trap from the point of view of obtaining an adequate vacuum. Furthermore, since air is continually leaking through the system, a moisture trap would require frequent replenishment. A rotary backing pump, retired from active service on really high vacuum processes, might well be used here. In this case the moisture trap could be omitted since moisture finding its way into such a pump, and so increasing the pressure of its ultimate vacuum, would not be an objection.

A vacuum gauge was also needed to enable the operator to adjust the pressure in the cathode ray tube to a suitable value.

Results. With these electrical and vacuum arrangements it was found possible to simulate electron bombardment from 5 kV to 10 kV by approximately a 40% change in gas pressure. The pressure registered on our gauge was of the order of 10^{-3} mm. Hg, although the exact pressure reading in such a dynamic system as this depends very much on just where the gauge is inserted.

This method of exciting the screen showed imperfections in the same manner as did an electron beam. With sample screens, which showed very differently under ultra-violet light, the r.f. excitation was almost indistinguishable from electrons.

Combination of Inspection with Aluminising Procedure

Since this accurate reproduction of flaws still applied to screens which had a cellulose film applied to them, it seemed possible to combine inspection with the aluminising process. This was tried out and found to be successful provided the evaporation filament allowed the screen an optical view of the aluminium sphere. (Fig. 4).

The procedure here was to pump the tube down to 10^{-3} mm. Hg and apply the R.F. power. If the screen passed this inspection, the air leak was closed, the pressure reduced to below 10^{-5} mm. Hg, and the aluminium could then be evaporated onto the screen.

Conclusions

A comparison of the appearance of several sample screens, under gas discharge at various pressures, and under electron bombardment at various anode potentials, showed that it is possible to simulate electron bombardment from approximately 5 kV to 10 kV.

The time required to evacuate 12 inch. and 14 inch. bulbs down to the necessary pressure for screen inspection was comfortably reduced to $2\frac{1}{2}$ minutes with the vacuum system described.

It was also found to be feasible to combine the aluminising filament with the exciting probe, and so to carry out both, aluminium evaporation and screen inspection, with only one pumping operation.

The method was found to be applicable to both, glass and metal bulbs, cellulosed and uncellulosed screens, and to glass bulbs with or without an internal aquadag layer connected to the anode terminal.

Perhaps of more academic than practical interest, is the way in which it may be demonstrated how great a protection against ionic bombardment an aluminium layer affords the screen. Once a tube has been aluminised the screen shows very little fluorescence on this apparatus, certainly not enough to be inspected for imperfections.

K. C. Burn.

Physical Laboratory,
Ferranti Ltd.,
Moston, Manchester 10, England.

14th March, 1955.

Vol
3
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BOOK REVIEWS

Zirconium. By G. L. Miller. (Butterworths Scientific Publications, London, 1954.) Pp. 382. Price £2 5s. 0d.

The element zirconium was isolated more than a century ago, but until comparatively recent years it has not been considered seriously as an engineering material. Like its sister metal titanium however, it has found widespread use for many years in the form of refractory compounds, vitreous enamels and in certain alloys. In the latter connection it has been added either as a scavenger in the manufacture of steel or as a grain-refining addition to non-ferrous alloys based on copper, nickel or magnesium.

With the rapid growth of the atomic energy industry, however, the industrial importance of hitherto rare metals has radically changed, and recent years have seen an intensive study of the extraction of zirconium from its ores and the development of satisfactory methods for the production of it in massive form at a very high level of purity. The rapidly-expanding zirconium industry presents an excellent example of the widening scope of modern chemical and metallurgical engineering and, if only for that reason, the present book by Dr. Miller makes fascinating reading. The publication, however, is timely because the present-day rate of progress in all branches of technology is so very rapid that a most useful purpose is always served by a monograph which authoritatively and concisely presents an up-to-date picture of developments in a specialist field.

Zirconium is widely distributed in nature and ranks eleventh in the list of elements in the earth's crust, which places it higher than that of common metals, such as copper, lead and zinc. The fact that it has not been more readily available in the pure form is due primarily to its great affinity for oxygen, and the complex problems involved in economically extracting it and converting it to the pure massive form. In the early chapters of this monograph, Dr. Miller outlines concisely the history and occurrence of zirconium, its use in refractory compounds, enamels and a limited range of engineering alloys.

Undoubtedly, one of the most important developments in the field of light metals has been that of the magnesium-zirconium alloys, which combine high strength and ductility in both the cast and wrought conditions. Alloys of this type, having excellent strength properties at moderately elevated temperatures, have played a significant rôle in recent years in developments in aircraft engines. The metallurgist will be disappointed that commercial alloys of this type are dealt with somewhat summarily, and out of proportion to their industrial importance.

The first stage in the preparation of pure zirconium is the decomposition of the ore, and the six more important methods used are described fairly fully, with no attempt to assess their relative merits and no indication of the preferred production processes. The methods of separation of hafnium from zirconium, so very necessary when the latter is required for certain nuclear applications, are outlined.

The basis of present day methods for the production of zirconium stem largely from the pioneering work of W. J. Kroll to whom, appropriately, this monograph is dedicated. Basically the Kroll process comprises the steps of carburisation of the zirconium mineral (*i.e.* zircon); chlorination of the zirconium carbide; purification and densification of the crude chloride; the reduction of the pure chloride with magnesium; separation of the reaction products from the reduced zirconium sponge. There have been, of course, developments in this basic process which have resulted in modifications facilitating the ease of manufacture and improving the economics thereof, and these are described quite fully. Dr. Miller is obviously very conversant with this aspect of zirconium metallurgy which is treated in a most authoritative and detailed manner. He does not hesitate to indicate future trends in the extraction process and, in particular, the possibility of the continuous working of the Kroll process by the reduction of the chloride and the melting of sponge in a combined continuous operation.

The van Arkel (*i.e.* the iodide) process in which the pure metal is produced by the dissociation of its iodide vapour on a hot filament is somewhat summarily dealt with in spite of the fact that large plants using this method are claimed to be in operation in the United States. Possible alternative methods of producing zirconium which are still under active investigation are described, such as, for example, the reduction of the tetrachloride by hydrogen; the reduction of the oxide by calcium or magnesium; the method based on disproportionation of the lower halides; the calcium reduction of the fluoride salt; and the electrolysis of fused complex chlorides and fluorides. Dr. Miller, refreshingly, does not hesitate to express his views in regard to the future developments of these methods, and dismisses most of them

as impracticable with the possible exception of the reduction of the fluoride with calcium and the electrolysis of fused salts. As in the case of titanium undoubtedly considerable scope still exists for development and improvement in production techniques, particularly in regard to a reduction in manufacturing costs.

The structure, the physical and mechanical properties, and mechanical and corrosion resisting properties of zirconium and zirconium alloys are described in some detail and occupy almost 100 pages of the text. In view of the significant rôle which the metal plays in the atomic energy industry, data on its nuclear properties and neutron absorption are of particular interest. The sensitivity of the metal with respect of oxygen, nitrogen and hydrogen is emphasized and, whilst this may have an important bearing on mechanical properties and corrosion resistance, it is a property of particular interest in electronics where ' clean-up ' of gases may be desired. The reaction of zirconium with liquid metals is covered somewhat tersely; its corrosion resistance is more fully described, and in this context attention is properly drawn to the all important general effect of metal purity. It is, perhaps, a little unfortunate that most of the mechanical property data are quoted for comparatively impure material. This, however, need not be taken as a criticism of the publication but rather an indication of the emphasis which has been placed upon the physical chemistry and metallurgy of the extraction of zirconium and its conversion to the massive form, rather than upon an investigation of its engineering properties.

The alloying behaviour of zirconium is discussed from the theoretical standpoint with the conclusion that, at the present time, modern alloying theory cannot be applied readily to zirconium alloy systems. Data on binary alloy systems, covering some 40 alloying elements, are described and, undoubtedly, this section alone is a most valuable addition to the library of any metallurgist, presenting as it does in a most concise and authoritative manner all the up-to-date information on binary zirconium alloy systems.

In the chapter on melting practice, Dr. Miller undoubtedly is very much at home with his subject and, obviously, has drawn extensively from his own considerable experience in this particular aspect of the zirconium manufacturing industry. His description of the various types of furnace which have been used for the melting of the zirconium sponge is particularly lucid and his review of the advantages and disadvantages of melting by induction, levitation, or in arc furnaces with a consumable or non-consumable electrode is clear and authoritative. Arc melting appears to be the favoured process, but it is clear that many problems still remain to be solved before a satisfactory melting procedure for zirconium can be established on a production basis. Judged by the enthusiasm which Dr. Miller evinces for his subject in this section of his monograph, there can be little doubt that the problems remaining will be investigated vigorously and successfully.

The fabrication of zirconium is dealt with in sufficient detail for it to be recognised that no very serious problems are present, provided that reasonable care is taken to preserve the purity of the metal, particularly with respect to contamination with oxygen, nitrogen or hydrogen.

The section on the powder metallurgy of zirconium is sufficiently comprehensive for it to be realised that the bulk of zirconium, undoubtedly, will be produced by melting and casting, although special cases will arise where sintered zirconium components will be required. Special compounds of zirconium, such as nitrides, borides, silicides and carbides, with an appendix on the metallography of the metal, round off the monograph.

It is inevitable that a monograph on a comparatively new engineering material will expose itself to a number of criticisms, particularly when it is written at a time when the technology of the metal is rapidly expanding. The layout of this monograph, in itself appears unnecessarily complicated and, undoubtedly, the reader will feel a little disappointed at the lack of information on certain developments which are known to be proceeding and which the monograph touches on rather lightly. However, Dr. Miller deserves the thanks of all metallurgists for having gathered together in one volume a most precise and authoritative description of a metal of growing engineering importance, and to have undertaken this task at a time when, undoubtedly, he realised that he was unable to disclose all of the technology with which he has been so closely associated.

I. JENKINS.

. . . of vacuum interest

Lectures

United Kingdom. THE ACTON TECHNICAL COLLEGE, HIGH STREET, ACTON, LONDON, W.3, announces a course of 12 lectures on BASIC HIGH VACUUM TECHNOLOGY on Fridays at 6.30 p.m. beginning on 13th January, 1956. The lectures will be given by D. J. Pacey, B.Sc., A.Inst.P. The course will be arranged as follows :—

1.—PROPERTIES OF GASES
Gas flow through tubes and orifices at low pressures. Application to design of vacuum systems.

2.—PUMPS AND SYSTEMS
Rotary pumps. Oil and mercury diffusion pumps. Assembly of complete high vacuum systems, with associated equipment.

3.—GAUGES
Various types in current use. Applications and limitations.

4.—LEAK DETECTION
Procedure and equipment for detection of leaks in systems.

5.—HIGH VACUUM MATERIALS AND PROCESSES
Vacuum tube assembly techniques, seals, electrodes, glasses. Properties and limitations of materials.

6.—HIGH VACUUM OPERATIONS
Evaporation of thin films of various metals and of high and low reflectance films. Sputtering of metals. Molecular Distillation. Freeze Drying.

Practical work will be based on topics selected from the above synopsis.

The fee is 2 guineas for the course. Further details are available from the Principal, Mr. J. Topping.

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United Kingdom. THE NORTHAMPTON POLYTECHNIC, ST. JOHN STREET, LONDON, E.C.1, announces a course on HIGH VACUUM TECHNOLOGY covering the fundamental principles of high vacuum production, technique and measurement. The course is held in two sections : (*a*) elementary and (*b*) advanced, and will include practical laboratory work. The course begins on Tuesday, 11th October 1955 at 7-9.30 p.m. The lecturer is J. Ewins, B.Sc.(Eng.)

United States. THE COMMITTEE ON VACUUM TECHNIQUES, formed last year, held its second Symposium at the Mellon Institute of Industrial Research in Pittsburgh on October 13th, 14th and 15th 1955. The following papers were read :

October, 13th, 1955

Welcoming address.
Presented by : *Dr. E. R. Weidlein,*
President of the Mellon Institute of Industrial Research.

Fundamental Developments

Application of Vacuum Techniques to Analytical Chemistry.
Presented by : *W. G. Guldner,*
Bell Laboratories.

Combined Tracer and Vacuum Technology for the Study of Micro-Weight Vacuum Distillation of Metals.
Presented by : *L. E. Preuss,*
Ford Institute for Medical Research.

The Technology of Large Mercury Pumped Vacuum Systems.
Presented by : *H. R. Smith,*
Radiation Laboratory, University of California.

A Graphical Solution for the Analysis of Vacuum System Performance.
Presented by : *D. J. Santeler,*
General Electric Company.

Explosions in Vessels Containing Hydrogen at Low Pressures.
Presented by : *E. G. Vogt & T. L. Robertshaw,*
Universal-Cyclops Steel Corporation.

Continuous Analysis of Gases in a High Vacuum Furnace with a Monitoring Mass Spectrometer.
Presented by : *H. Landsberg, E. E. Escher & Dawkins,*
Consolidated Engineering Corporation.

Recent Trends in Vacuum Deposition of Precision Optical and Electrical Films.
Presented by : *R. A. Denton & T. L. Scatchard,*
Optical Film Engineering Company.

October 14th, 1955

Applications

Applications of Vacuum Techniques to Metallurgical Research.
Presented by : *R. F. Dickerson,*
Battelle Institute.

Recent Innovation in Vacuum Metallurgy.
Presented by : *G. J. Crites,*
F. J. Stokes Company.

High Vacuum Impregnation of Oil Filled Capacitors and Cables.
Presented by : *F. W. Oetjen,*
Leybold Nachfolger.

Television Tube Aluminising
Presented by : *S. W. Kennedy,*
National Research Corporation.

Vacuum Dehydration of Refrigerator Components.
Presented by : *H. A. Steinherz*,
National Research Corporation.

Development and Performance of a New Ion Gauge Tube and Control Circuit.
Presented by : *J. Peters*,
Vacuum-Electronic Engineering Company.

October 15th, 1955

Methods and Techniques

Evapor-Ion Pump Performance with Noble Gases.
Presented by : *I. Alexeff & E. C. Peterson*,
University of Wisconsin.

Evapor-Ion Pump Characteristics.
Presented by : *J. Swartz*,
Consolidated Vacuum Corporation.

Standards

Standards of Performance Ratings for Vapour Pumps.
Presented by : *B. B. Dayton*,
Consolidated Vacuum Corporation.

Standards for Helium Leak Detector Calibration.
Presented by : *B. B. Dayton*,
Consolidated Vacuum Corporation.

A Banquet was held on October 14th at the William Penn Hotel where the guest speaker, Mr. Willy Ley, spoke on ' The Physics and Engineering of a Satellite Shot.' Further information on the Symposium can be obtained from : Committee on Vacuum Techniques, Box 1282, Boston 9, Mass., U.S.A.

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France. THE SOCIETE DE CHIMIE-PHYSIQUE will hold a series of discussions on SURFACE REACTIONS OF GASES ON METALS on the occasion of their 6th Annual Meeting which will take place on the 22nd-25th May, 1956. The discussions will essentially deal with the fundamental phenomena which determine the structure of thin films formed by the reaction of gas at a metal surface. Adsorption and catalysis will not be included. Further details can be obtained from : Secrétaire Général, Société de Chimie-Physique, 10 rue Vauquelin, Paris (5c).

FURTHER LECTURES are announced as follows :

September 12th, 1955, *at 2 p.m.*

Basic Elements for Pressure Control.
Lecturer : *A. M. Thackara.*
Held at : Shrine Auditorium, Los Angeles, California, U.S.A.
Arranged by : Instrument Society of America (1955 ISA Conference).

September 15th, 1955, *at 2 p.m.*

A Method for Continuous Subatmospheric Sampling for a Process Monitor Mass Spectrometer.
Lecturers : *E. E. Escher & H. Landsberg.*
Held at : Shrine Auditorium, Los Angeles, California, U.S.A.
Arranged by : Instrument Society of America (1955 ISA Conference).

October 6th, 1955, *at 7.15 p.m.*

Vacuum Melting and Vacuum Heat Treatment.
Lecturer : *B. C. Woodfine.*
Held at : Large Chemistry Theatre, University of Leeds.
Arranged by : Institute of Metals and the Leeds Metallurgical Society.

October 6th, 1955, *at 7 p.m.*

Development of the Lost-Wax Process of Precision Casting.
Lecturer : *J. S. Turnbull.*
Held at : Keighley.
Arranged by : Yorkshire Section, Institution of Mechanical Engineers.

October 25th, 1955, *at 6.45 p.m.*

Metallic Coating of Plastic Mouldings.
Lecturer : *L. Holland.*
Held at : Bell Hotel, Leicester.
Arranged by : Midlands Section, Plastics Institute.

Licensable Patents

THE NATIONAL RESEARCH DEVELOPMENT CORPORATION* which is responsible for the exploitation and where necessary, development of inventions received from the expenditure of public funds (see Vol. II No. 3) has sent us particulars of further patents as follows :—

Pat. Appln. No. 12030/50. *Heating Element for Use in a High Vacuum Furnace.*

The invention is a resistance heater in the form of a sheet metal strip end-supported by current conductors, a portion of the strip near the centre of the strip length being longitudinally curved or bent to form a heating cavity, and the remainder of the strip being repeatedly folded back and curved or bent to form a succession of sheaths around the heating cavity and spaced apart from each other for limiting the heat loss. In one form, the strip is tapered from its ends to its centre to give a very high temperature heating cavity. In another form, elements according to the invention are used to form a furnace comprising two

* Enquiries can be addressed to : Dr. B. J. A. Bard, Industrial Manager, The National Research Development Corporation, 1, Tilney Street, London, W.1, England. (Tel. : Grosvenor 5431.)

or more such elements co-axially arranged so that the end of one element laps the end of an adjacent element.

Pat. Appln. No. 12065/53. *Apparatus for Feeding Filamentous Material Continuously to a Vapourising Element in Vacuum Apparatus.*

The invention provides a means for feeding aluminium wire through a stainless steel scanning guide tube on to a heating element, where the wire is vaporised. The scanning guide tube is provided with a means to oscillate so as to scan repeatedly along the line of the vaporising element, and a means for driving filament material through the guide tube. The apparatus can be used in conjunction with vaporising elements as described in Pat. Appln. No. 10827/52 (see Vol. II No. 3, p. 266).

Pat. Appln. No. 20411/53. *Foil Feed Mechanism for Continuous Vaporisation of Metals.*

The vacuum vaporisation apparatus comprises a linear vaporising element capable of spreading material fused thereon substantially uniformly, a means for feeding material to be vaporised in the form of a thin sheet or foil so that the advancing edge of the sheet extends along the elements, and a means for controlling the feed rate of the material such that the material fuses over the element before being vaporised. It is preferable that the thin sheet or foil to be vaporised is drawn from a roll and fed through a slot in a reflector plate associated with the vaporising element. The element described in Pat. Appln. No. 10827/52 is suitable (see Vol. II No. 3, p. 266).

Pat. Appln. No. 22968/53. *Rotating Turret for Vaporising Element in Vacuum Vaporisation Apparatus.*

The invention is a device whereby a series of vaporising elements can be brought to bear upon a target in sequence so that it is possible to vaporise a larger quantity of material than that available from a single element or so that a series of dissimilar layers may be vaporised on to a target without letting down the vacuum in the apparatus. The device consists of a series of rod-like vaporising elements each having a helical rib along their length cylindrically arranged on a rotating holder and electrical contact means for heating the elements in turn as the turret is rotated.

Pat. Appln. No. 19109/54. *Methods for Controlling the Deposition of Dielectric Layers on Interference Filters.*

This invention is a development of the method used by Giacomo and Jacquinot for very accurate layer thickness control. This is achieved by exploring the filter during actual deposition of each layer by a light beam so modulated that its wavelength exhibits progressive or abrupt alternations between the extremes of a small range. Deposition is terminated when immobility of the transmission has a stationary point at a specified wavelength. This indicates that the layer thickness is correct for this wavelength.

Pat. Appln. No. 5486/55. *Method of Optical Aspherising by Evaporation.*

By using suitably shaped rotating masks a layer of zinc sulphide may be deposited up to the order of 100 wavelengths in thickness to form aspheric surfaces. Zinc sulphide has previously been regarded as unsuitable for this purpose as, owing to its high refractive index, light losses due to surface reflection are unacceptably high. The use of specially devised double layer antireflection blooms between the base and the zinc sulphide, and on the outer surface of the latter is described.

Professional

September, 1955

United Kingdom. The Institutions of Civil, Mechanical, Electrical and Chemical Engineers and the Institute of Physics have formed a new body, the *British Nuclear Energy Conference* with Sir Christopher Hinton as the Chairman and Mr. Alexander McDonald as the Secretary. The new body is to concentrate on the presentation and dissemination of information on nuclear energy subjects in the form papers or discussion meetings. Also, the Conference will publish a quarterly periodical under the title of *The Journal of the British Nuclear Energy Conference* commencing in January 1956. Further information can be obtained from the Secretary, British Nuclear Energy Conference, 1-7, Great George Street, London, S.W.1.

Personal

October, 1955

United States. FRANCIS J. STOKES, founder and chairman of F. J. Stokes Machine Company died recently in Philadelphia. He was 81 and had been for 53 years at the head of the Company.

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Books

The following books on vacuum subjects have been published during the last few months (1955):

Germany

Wolfgang Gaede—Der Schoepfer des Hochvakuums.
Author : *H. Gaede*
Published by : Verlag G. Braun, Karlsruhe.
Pp. 127. Price 9s. 6d.

Hochvakuum Technik
Author : *J. Yarwood* (*Translator Dr. H. Adam*)
Published by : Rudolf Lang Verlag, Berlin-Charlottenburg
pp. 208. Price £1 5s. 0d.

United Kingdom

High Vacuum Technique (3rd revised edition).
Author : *J. Yarwood*
Published by : Chapman & Hall Ltd., London.
pp. 208. Price £1 5s. 0d.
(*to be reviewed*)

Ionised Gases
Author : *A. von Engel*
Published by : Oxford University Press, London
pp. 292. Price £1 10s. 0d.
(*to be reviewed*)

Industrial

June, 1955

Italy. EDWARDS HIGH VACUUM LTD., in association with Societa Apparecchi Elettrici e Scientifici, has formed an Italian company under the name of Edwards Alto Vuoto S.p.A., with offices at 84, Via Monte Rosa, Milan. Dr. P. Della Porta is the Managing Director of the new company.

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July, 1955

United Kingdom. As recently published in the daily press Imperial Chemical Industries Ltd. have started producing titanium at a new plant the output of which will be 1,500 tons a year.
Germany. Messrs. Krupp at Essen are producing titanium at a rate of 36 tons per annum.
The present total World Production of titanium is estimated to exceed 5,000 tons per annum. This excludes Eastern Europe.

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On the Subject of Pinholes :

The Inspection Department: "*Too many pinholes in this mirror.*"

The Production Department: "*This is a mirror, not a peep show—measure the reflectivity.*"

VACUUM
CLASSIFIED ABSTRACTS

Vol.
3
1953

Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

Arrangement of Subjects

All abstracts published in this section are allocated to one or more of the subjects shown in the following list:

(I) General Science and Engineering

10 — VACUUM ENGINEERING — GENERAL
11 —
12 —
13 — ELECTRICAL SCIENCES
14 — MECHANICAL SCIENCES
15 — PHYSICS
16 — CHEMISTRY
17 — METALLURGY
18 —
19 — SCIENCES (other than the above)

(II) Vacuum Apparatus and Auxiliaries

20 — PUMPS
21 — GAUGES
22 — MEASURING PLANT — VACUUM
23 —
24 —
25 —
26 — MATERIALS
27 —
28 — APPARATUS COMPONENTS
29 — MISCELLANEOUS APPARATUS

(III) Vacuum Processing Techniques

30 — EVAPORATION
31 — CATHODIC SPUTTERING
32 —
33 — IMPREGNATION
34 — FUMIGATION
35 —
36 — DRYING
37 — METALLURGICAL PROCESSES
38 — DISTILLATION
39 — MISCELLANEOUS PROCESSES

(IV) Special Subsidiary Subjects

40 — GASES & VAPOURS
41 — FREEZE DRYING
42 —
43 —
44 —
45 — ELECTRONICS
46 —
47 — GLASS PROCESSING
48 —
49 —

Indices

Three indices are appended to each issue of abstracts: (1) A Cumulative Subject Index in front of the text, (2) a Cumulative Author Index at the back of the text and (3) a List of Periodical Publications quoted. Index (1) and (2) will show all abstracted information published up-to-date in the current volume of the journal.

Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract, *e.g.*, 20/I as distinct from 20/II, etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

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I — General Science and Engineering — I

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10 — VACUUM ENGINEERING (GENERAL) — 10

A New Vacuum Society 187/I

United Kingdom. In a recent issue of this journal it was tentatively suggested to form a vacuum working group under the auspices of the various interested professional institutions in this country. The author of the present letter supports this suggestion and states that in his experience the development of vacuum technology has reached a stage, where the existence of such a working group would be of decided advantage to further progress.

Sommaire: Dans cette lettre référence est faite de l'éditorial du premier numéro du Volume No. III de ce journal, et on a conseillé de former un groupe d'étude du vide.

Letter by D. W. Stops *Vacuum* 3, July 1953 288

Descriptive Terms for Vacuum Fumigation 188/I

United Kingdom. It is suggested that the terms normally used to designate the various techniques employed in vacuum fumigation should be altered to give a more accurate description. There are four different methods: (1) The pressure in the chamber is reduced. Subsequently the fumigant is introduced. The vacuum is maintained for the whole length of the treatment. This method is known as ' sustained vacuum ' fumigation. No change of name is suggested. (2) The pressure in the chamber is raised to atmospheric immediately after introduction of the fumigant and remains at this value for the rest of the processing time. This method, hitherto known as ' released-vacuum ' method would be better described by ' vacuum fumigation with atmospheric pressure restored immediately after dosage '. (3) After admission of a mixture of air and vaporised fumigant, the chamber pressure is raised to atmospheric. This, the so-called ' French released-vacuum ' method should be termed ' vacuum fumigation with simultaneous admission of air and fumigant '. (4) After admission of the fumigant, the reduced pressure is maintained for a while; then air is admitted and treatment continued at atmospheric pressure. This method should be known as ' vacuum fumigation with atmospheric pressure restored after a period '. It is questionable whether the general term ' vacuum fumigation ' is adequate as the operational pressures are never lower than 5 cm. Hg but no change of term is proposed.

Sommaire. On suggère que certains termes utilisés en fumigation sous vide sont ambigus et des nouveaux termes d'un caractère plus définitif sont récommendés.

Letter by A. B. P. Page R. E. Blackith W. Burns-Brown & S. G. Heuser *Chem. & Industr.* 11, April 1953 353-354

Vacuum Technique 189/I

United Kingdom. Recent progress in vacuum equipment design and application is surveyed. The following new books are mentioned : ' Vacuum Technique ' by Reiman, ' Freezing and Drying ' by Harris (Editor) and ' Miscellaneous Physical and Chemical Techniques ' by Snyder and Lipkin. The author describes a rotary pump attachment for the removal of volatile solvents entrained in the pump oil. The oil is brought to the top of a pipe, 3 ft. high and 4 inch. in diameter, by means of a special gear pump. From there it flows downwards over a steam-heated copper coil which causes the volatiles to evaporate. After cooling the oil is returned to the rotary pump. Some details are given of a newly developed self-cleaning fractionating three-stage diffusion pump. Various gauge developments are discussed. The new design of a rotary McLeod gauge by Axelbank is described and shown in a sketch. The gauge is made of glass and covers the range of 10^{-2} to 10^{-6} mm. Hg. The circuit of the standard alphatron gauge has been redesigned to facilitate its use in conjunction with a recorder unit. A similar gauge, especially developed for use in corrosive gas mixtures, has a polonium-on-nickel source and covers the range of 0.1 to 100 mm. Hg. A tetrode ionisation gauge with two grids and a thin silver ion collector on the glass wall has been proposed by Warmholtz which has a range down to 10^{-8} mm. Hg. Some details are given of Leck's investigation into the performance and range of the Pirani gauge. (See Vol. II Abstract No. 132/II.) New developments in vacuum plumbing are mentioned. For example, Knudsen has found that 1/16 inch rings, made from indium sheet 0.007 inch thick, make efficient seals between metal and other surfaces and can be used in place of gaskets made from organic materials, where use of the latter is undesirable. However, on opening the seal the ring is destroyed. Stanier's design of a greaseless vacuum stopcock is described and shown in a sketch (See Vol. II Abstract No. 108/II.). Several vacuum processing methods in metallurgy are briefly described with particular reference to molybdenum, tantalum, titanium and zirconium. Vacuum arc-furnaces working on the consumable-electrode principle have been built to give molybdenum ingots of up to 1,000 lb. with an oxygen content of less than 0.003%. Vacuum-sintering tantalum by passing current through tantalum powder pressed into bars at temperatures of 2,500°C in a furnace continuously pumped at 10^{-4} mm. Hg, produces a material which can be rolled into sheets. It may be necessary to vacuum-anneal the sheets at 1,500°C. The production of titanium, believed to be permanently dependent on vacuum methods, increases steadily. The largest ingots cast to date are 2 tons. Details are given of the final stages of zirconium production, freeing the zirconium sponge from excess magnesium and magnesium chloride.

Sommaire: Un article de portée générale sur les applications de la technique du vide portant en particulier sur les modèles de pompes, d'appareils de mesure et sur le developpement du traitement des métaux sous vide.

Article by S. L. Martin *Chem. Process Engng.* 34, Sept. 53 276-279

Apparatus for Evacuating Hollow Articles 190/I

See Abstract No.: 173/II

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Abstract No. and References

New Developments in the Production and Measurement of Ultra High Vacuum

191/I

United States. The paper deals with devices and instruments developed for the production and measurement of vacua below 10^{-8} mm. Hg, the ultimate pressure normally obtained with diffusion pumps. In theory the ionisation gauge should indicate pressure as low as 10^{-16} mm. Hg but in practice the standard design prior to 1948 did not indicate pressures lower than 10^{-8} mm. Hg. It was proved, however, that lower pressures were, in fact, obtainable already at that time. Nottingham suggested in 1947, that at this pressure level, a residual current, caused by soft X-rays and created by electrons striking the grid in the gauge, flows to the ion collector which makes it necessary that the value of the ion current is comparatively high in order to give a proper indication on the gauge. Apker in 1948 extended the range of the gauge by using the ' flash filament ' method which provides for an auxiliary tungsten filament to be added to the gauge which is periodically flashed at temperatures above 1,500°K. During flashing, the gas molecules adsorbed on the filament between two flashes are released and cause a momentary peak pressure indication on the gauge. The time required to cover the filament with a monolayer of gas between two flashes can be ascertained and serves as a measure of the pressure prevailing in the vessel. The Bayard-Alpert ionisation gauge, first described in 1950, was specially developed to overcome the X-ray effect. In the new gauge the filament is outside the cylindrical grid and the ion collector, a very fine wire, is suspended within the grid. The ion collector, no longer cylindrical, presents a solid angle to the X-rays from the grid, about a thousand times smaller than in previous designs. The ion collection efficiency is about the same as before. The range of the new gauge extends to 5×10^{-11} mm. Hg. It was found that the ratio of the residual currents in the conventional and the new gauge is equal to the ratio of the aforementioned solid angle presented to X-radiation. Measurement with the help of ionisation gauges has two further limitations : (*a*) A varying sensitivity for different gases. (*b*) The ' clean up ' action of the gauge. In operation, the gauge removes gases by chemical and by electrical action, *i.e.* molecular gases may be dissociated at the hot cathode and the atomic substances combine at the gauge walls and the accelerating potentials applied to the electrodes may cause ions to be trapped in the collector. The theoretical aspect of the pumping action is considered on the base of a previous investigation by Schwarz and the following conclusions are arrived at : The gauge can be used to pump sealed systems and the characteristic pumping times for common gases can be determined. If used for measurement, the gauge should be operated for short periods only, not to alter the pressure conditions in the vessel. The design of a bakable valve is described and shown in illustrations which holds an ultra high vacuum in a one-litre system with a leakage conductance of about 10^{-11} l./sec. It consists of the valve assembly, to be incorporated in the vacuum system, and the driver mechanism. The former contains three machined parts. A copper cup, $1\frac{3}{4}$ inch. diameter with $\frac{1}{4}$ inch diameter holes for the vacuum orifices, a flexible Kovar diaphragm allowing an up and down movement of about 0.1 inch and a Kovar nose seating in the copper cup. The seal is formed, when the valve is closed the first time, by forcing the nose into the vacuum orifice in the copper cup. The driver mechanism is detached prior to baking and consists either of a differential screw mechanism or of a direct screw thrust mechanism, exerting a force of 1–2 tons. It follows the description of an absolute manometer, suitable for use in conjunction with ultra high vacuum systems which has been reported previously (see *Rev. Sci. Instrum.* **22** 1951, 370). The development of the elements discussed above facilitated a new approach to the design of vacuum systems. Details are given of a method which provides for the system to be made up of a series of module units, each 16 inch. square, which fit standard furnaces, single or in pairs, for baking out. Each furnace unit has eight heaters requiring 1 kW to produce 450°C. In some instances systems of this kind were installed by the glassblower in the course of one morning, exhausted by diffusion pumps and checked for major leaks in the afternoon, baked for 8 hours overnight and subsequently pumped with the help of ionisation gauges reaching the ultimate of 10^{-10} mm. Hg by noon of the second day. The author concludes the article with a discussion of suitable leak detection methods and the description of an ion gauge control circuit providing for intermittent gauge operation.

Sommaire: Une discussion est faite au sujet de plusieurs nouveaux appareils pour la production et la mesure du vide extrèmement poussé (10^{-10} mm. Hg.).

Article by D. Alpert *J. Appl. Phys.* **24**, July 1953 860-876

A New Manometer for the Pressure Region of 10^{-2} mm. Hg to 10^{-6} mm. Hg

192/I

See Abstract No.: 141/II

Isolation Valves for Vacuum Systems

193/I

See Abstract No. : 163/II

A Vibrating Needle Variable Gas Leak

194/I

United States. The sketch is shown of a solenoid-operated needle valve originally designed for the admission of bursts of gas to the chamber of an electron diffraction camera. The needle is fitted to a shaft, which is fixed to the bellows-supported topplate of the valve housing. The ' atmospheric ' end of the shaft carries a steel core which is surrounded by a magnet coil fitted to a separate metal frame. The length of the stroke of the shaft can be controlled by an adjusting screw in the metal frame. Applying a.c. current the needle will oscillate in an axial direction permitting the admission of gas in any desired quantity. At the pressure level of 10^{-5} mm. Hg pressure changes in the vacuum system can be controlled to a very fine degree, limited only by the minimum indication obtainable from a standard ionisation gauge.

Sommaire : Description est faite d'un modèle de vanne à aiguille controlée électriquement dont le but est de permettre l'admission d'un volume précis de gaz dans la chambre à vide d'une camera à diffraction électronique.

Note by A. J. Stinnett *Rev. Sci. Instrum.* **24**, Sept. 1953 883-884

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Abstract No. and References

A Leak Control Tube 195/I

See Abstract No. : 165/II

Electrode Contamination in Electron Optical Systems 196/I

United Kingdom. It is well known that black deposits and high resistance films showing interference colours tend to form on the electrodes in a demountable vacuum system and cause instability of the electron optical system. The nature and formation of these deposits which originate from the materials used in joints has been investigated. A mercury diffusion pump and a liquid oxygen trap was used for evacuation in the experiments, the joint under test being in a position between trap and system. The vacuum system was of glass. In order to measure the breakdown temperature of the films produced by a low energy electron beam, a target electrode which could be heated by radiation was exposed to the beam and subsequently pivoted by magnetic means into a position where it came into contact with a copper electrode and the resistance between target and copper electrode was measured. The method of estimating the temperatures is given. Using greased joints and a beam of 2mA at 100V for 20 hrs. the following values were found: Conductivity of the deposit—approx. $10^{-9}\Omega^{-1}cm^{-1}$, breakdown temperature in vacuum—approx. 600°C and breakdown temperature in air—450°C. These results are consistent with values obtained by Koenig and Helwig for the case of a gas discharge in organic vapours, indicating identity of the deposits obtained. Ions formed in organic vapours may lose their charge and form free radicals which may either decompose further giving ' molecules ' consisting of a carbon chain with occasional hydrogen atoms attached or may polymerise into stable long-chain molecules. In order to obtain further confirmation that the deposits in the present experiments are of an identical nature, further experiments were carried out, details of which are given proving that free radicals were present. Investigating the distribution of the deposits in a special experimental arrangement, shown in a sketch, evidence was found suggesting that free radicals can be formed without an intermediate ionisation stage. After having established the nature and mechanism of formation of the contaminating films, the author investigated the effects obtained from different sealing materials and from a variation of beam intensity. Various target materials were used in the case of Apiezon grease as the sealing material. The results were similar for all target materials. No high-resistance films were formed in a beam at energies exceeding 300V. Contamination could be prevented altogether by heating the target to 250°C during exposure to the beam. Everett's No. 1 wax, de Khotinski and Araldite cement behaved similarly to Apiezon grease. Silicon, grease and Picein wax gave the highest resistance values. The only sealing material which gave no high-resistance films at beam energies below 300V, was Neoprene.

Article by K. M. Poole *Proc. Phys. Soc.* **66B** July 1953 542-547

Sommaire : On a fait des recherches sur l'origine, la consistance, la structure at les effets des films parasites sur l'électrode d'un appareil optique électronique.

Cathodic Sputtering—an Analysis of the Physical Processes 197/I

See Abstract No. : 153/III

Reactive Sputtering and Associated Plant Design 198/I

United Kingdom. The technique, termed *reactive sputtering*, of preparing thin films of metallic compounds, notably metal oxides, by sputtering from metallic cathodes in the presence of active gases is described and compared with other deposition processes. The oxidation mechanisms which may occur during the sputtering of a metal in an atmosphere containing oxygen are considered. The desirable performance features of a reactive sputtering plant are enumerated. Experience of producing a range of metal oxide coatings has shown that in order to obtain films of a specified chemical composition the concentration of the desorbed gases in the sputtering atmosphere must be reduced to a minimum. This can be achieved by the use of a vapour pump, having a sufficiently high speed at the sputtering pressure to permit a high rate of leakage of the desired gas into the sputtering chamber. The composition of films of metal oxides which do not obey the law of fixed proportions can be varied by sputtering in oxygen plus inert gas mixtures of different concentrations. A simple method for controlling the composition of such gas mixtures is described. Consistent control of the sputtering rate requires the provision of a stable glow discharge operating at a given applied voltage and current density. It is shown that a booster vapour pump used in conjunction with a high vacuum isolation valve of variable aperture permits fine control of the sputtering pressure on which the discharge voltage and current are dependent, also, that a stable glow discharge can be easily obtained if the high-tension transformer is of the high reactance type.

(*Authors*)

Article by L. Holland & G. Siddal *Vacuum* 3, July 1953 245-253

Sommaire : Description est faite des principes de le projection cathodique réactive c.a.d. projection cathodique par cathodes métalliques en présence de gaz actifs. Une discussion est faite sur la réalisation d'une machine á projection cathodique réactive.

An Electro-Micrograph Study of Oxide Films on Electro-Polished Surfaces of Iron 199/I

See Abstract No. : 250/I

Molecular Distillation 200/I

See Abstract No. : 175/III

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The Frozen Bone Bank of the Netherlands Red Cross 201/I

See Abstract No. : 68/IV

Freeze Drying and the Tissue Bank 202/I

See Abstract No. : 243/I

Theory of the Vacuum Drying of Frozen Tissues 203/I

United States. The results of research work into freeze drying of blood plasma and materials with a small solid component do not apply generally to the drying of tissue without some modification. One factor of major importance in tissue drying is the effect of the dry shell which developes as the tissue dries. A theoretical and experimental analysis of this effect is given in this paper. During freezing of the tissue, ice crystals of a size up to several microns are formed, each of which is surrounded by ' walls ' of the materials to be dried. In this manner the well-known honeycomb structure of the fully dried material is originated. As the drying proceeds the level of the residual moisture content recedes into the interior and a dry shell, ever growing in thickness, is formed. Sublimation of the ice takes place at the interface of the dry shell and each evaporating molecule has to find its way through the honeycomb structure of the shell in order to reach the drying chamber and eventually the condenser or the trap. The dry shell develops into a formidable obstruction as can be seen from the following consideration : If the mass of the subliming molecules which cross the unit area of the (assumed) horizontal interface in a perpendicular direction is $P/(2\pi RT)^{\frac{1}{2}}$, where P is the pressure of the vapour, T the absolute temperature of the vapour and R the gas constant per gram., then the net flow per unit area through a dry shell, 1,000 layers of honeycomb cells thick, is expressed by

$$\frac{dm}{dt} < \frac{1}{1{,}000} \frac{P_s}{(2\pi RT_s)^{\frac{1}{2}}} \qquad (1)$$

where the suffix s indicates the conditions at the subliming surface. Assuming that the average ice crystal formed has a size of 1 micron, this corresponds to a dry shell of 1 mm. thickness which reduces the rate of drying by a factor of approx. 1,000. In order to obtain information more directly useful to the designer of drying apparatus, some indication of the drying efficiency in terms of the fraction of escaping molecules is required. For this reason the relation between vapour flow and heat flow is considered, introducing the factor of escape probability, f. The net rate of evaporation is then given by

$$\frac{dm}{dt} = \frac{(f \alpha P_s)}{(2\pi RT_s)^{\frac{1}{2}}} \qquad (2)$$

where α is the coefficient of evaporation. For $f=1$ this formula gives the maximum rate of sublimation at any given temperature of the ice with no obstruction in the path of the subliming molecules. The factor f is made up of two components : f_e representing the fraction of molecules which have reached the drying chamber and which are condensed or trapped before they are recaptured by the processed material and f_s representing the fraction of molecules which have reached the drying chamber without being stopped inside the drying material. Thus, assuming that all molecules which reach the drying chamber are condensed, *i.e.* that $f_e=1$ and substituting f_s for f, Equ. 2 gives the maximum evaporation rate in the presence of a dry shell. The net rate at which heat has to be supplied to the unit area of the interface between the dry shell and the frozen material in order to facilitate sublimation is expressed by

$$\frac{dQ}{dt} = L \frac{dm}{dt} \qquad (3)$$

where L is the latent heat of sublimation per gram of ice. Thus it can be stated

$$\frac{dm}{dt} = \frac{\alpha f_s P_s}{(2\pi RT_s)^{\frac{1}{2}}} \cdot \frac{1}{1 + f_s/f_e - f_s} = \frac{K}{L} (T_c - T_s) \qquad (4)$$

where K is the general heat transfer coefficient and T_c the temperature prevailing in the immediate neighbourhood of the interface. This is the basic formula for the determination of f_s, the fractional drying rate, which governs the design of drying apparatus. K/L is a function of the thickness of the dry shell. The time needed to dry a shell of the thickness α is given by

$$t = \int_0^x w \frac{(2\pi RT_s)^{\frac{1}{2}}}{\alpha P_s} \left(\frac{1}{f_s} + \frac{1}{f_e} - 1 \right) dx \qquad (5)$$

where w is the ice content in g/cm³ of the frozen tissue. The above theory has been used for the determination of the drying rate of guinea pig liver in a special series of experiments. Details of the apparatus employed and of the results obtained are given. It was experimentally confirmed that at a given ice temperature the drying rate of the liver was smaller by a factor of 1,000 compared with the sublimation time of ice of the same size.

Sommaire : Une analyse de l'effet produit par la couche sèche d'une matière dans le procedé de lyophilisation est donnée.

Paper by J. L. Stephenson *Bull. Math. Biophys.* 15, 1953 411-429

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A New Method of Preserving Fresh Human Milk 204/I
See Abstract No. : 69/IV

Vacuum Pasteurisation 205/I
See Abstract No. : 185/III

13 — ELECTRICAL SCIENCES — 13

Induction-Melting Furnaces 206/I
See Abstract No. : 257/I

Crystallographic Variations of Field Emission from Tungsten 207/I
See Abstract No. : 79/IV

The Vacuum Technique of the Mercury Vapour–Iron Rectifier. Part I 208/I

Germany. The first mercury vapour rectifiers were built some 50 years ago. At a very early stage in their development iron was substituted for glass as construction material for the vacuum chamber. Initially the designers favoured the development of high capacity units fitted with large pumping plant comprising mercury diffusion pumps, mechanical backing pumps, gauges and automatic controls. However, the high maintenance cost, the heavy weight of the apparatus and uncontrollable losses due to the large dimensions involved, caused the development of smaller units during the years prior to the war. At the same time attention was given to methods of improving leak-tightness of the vacuum chamber and to methods of sealing-in the electrical leads which would permit proper de-gassing of the assembly. As a result the multiple-anode pumpless rectifier and the single-anode rectifier of the ignitron type, became available. The action of the mercury vapour iron-rectifier is based on the one-way valve effect of the mercury vapour. The capacity of the apparatus is governed by the maximum value of the current, at which the mercury vapour still exerts this effect safely and this, in turn, is controlled by the quality of the vacuum prevailing in the chamber, *i.e.* by the residual pressure of gases and vapours other than the mercury vapour in the chamber. When not in use, the pressure in the chamber of the rectifier should not exceed 10^{-3} mm. Hg. The operational pressure is 10^{-2}–10^{-1} mm. Hg. Continuously pumped rectifiers should be fitted with pumps which are capable of exhausting to 10^{-5} mm. Hg. Iron is used as a construction material because it does not react with mercury. Its permeability for oxygen is negligible but its permeability for nitrogen at temperatures beyond 250°C is excessive. The rate of permeation of hydrogen is 10^6 greater than that of nitrogen and is particularly dangerous where permeation takes place in the form of H^+ ions originating from the cooling water, to be released as gaseous H_2 into the chamber. This effect is absent, where chromium-iron alloys or other non-corroding iron alloys are used. Various designs of current lead-ins are described. In the case of demountable constructions flanged joints are used, employing a porcelain ring as intermediary component. The actual seal can be effected by aluminium rings, which are coated with a layer of plastics material on one side. This ensures good adhesion even if the aluminium ring should be distorted after assembly. Permanent seals are effected by glass-to-metal seals, such as tubular lead-ins of Kovar material sealed to glass, frequently employed in ignitrons and permitting bake-out temperatures up to 400°C. Another method provides for a ceramic-glass-metal seal, where Steatite is used as the ceramic and an iron-nickel alloy as the metal. A leak testing method originally introduced by Dallenbach is described : Ammonia is used as the testing gas and covers saturated with mercurous nitrate, spread over the areas to be tested, are used as indicators. In place of mercurous nitrate, dyes may be employed, the colour shades of which depend on the pH value. Leaks down to 10^{-5} mm. 1/hr. can be traced with this method. To counteract the evolution of gas during operation the chamber is de-gassed at 300–400°C. This is the temperature range just above the point, where the liberation of gases from iron is at a maximum. Pumpless rectifiers de-gassed in this manner have a residual gas pressure of less than 10^{-3} mm. Hg even after several years. Degassing by the normal operational electric arc initiates gas reactions which result in the liberation of carbon and the formation of the characteristic ' soot layers ' often observed in rectifiers. The presence of these layers may lead to parasitic discharges which break up the layers, cause the carbon to deposit on the anode thus reducing the working area of the electrode and resulting in excessive current density which will eventually destroy the valve action of the mercury vapour.

Sommaire : Des détails sont donnés sur les modèles et l'action des redresseurs à vapeur de mercure.

Article by G. Dobke *Glas & Hochvakuum Tech.* **2**, Sept. 1953 249-254

Microstructure of Capacitor Paper 209/I

United States. Kraft capacitor paper is manufactured from cellulosic wood pulp. It is available in thicknesses from 0.0002 inch upward and is normally subjected to an electrical stress of 800V/mil at operational temperatures up to 100°C. The chemical composition of the paper and the effect of any chemical variations on the performance have been studied thoroughly. But studies of the physical properties of the paper were so far

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confined essentially to porosity, density and the nature of the conducting particles. Kraft paper is known to show a pronounced fibre structure. It was the object of the investigation reported here to investigate the physical features of the fibres. Fibres, in the interpretation given by the authors, are not ' native ' fibres but materials present in filamentary form as distinct from such materials as resins, amorphous agglomerates and non-filamentary fibre debris. Non-uniformity of paper thickness is believed to be due to the dimensions of the fibres. Investigating 0.4 mil (10 micron) paper under the light microscope showed that there were only a few spots with a thickness of 1 mil (25 micron) in paper which contained fibres of 25 micron width. Examination of the sample before and after calendering proved that the fibres were not cylindrical or rod-shaped but flat and ribbon-shaped. In order to confirm this, specimens of 0.4 mil paper were examined in the electron microscope. The method of preparation is given. Silica was evaporated on to a plastic mould of a microscope slide surface and the fibres deposited on the silica substrate from a suspension. The specimen was shadow-cast at an angle of 53.5° so that the shadows cast were twice as long as the respective elevations. It was established by these studies that the fibres were ribbon-shaped with a thickness of 0.01–1 micron. Occasionally the fibres were twisted and stood on edge over a certain length. Other features observed in these experiments were : (*a*) The presence of ' colonies ' of granular material which formed when the liquid medium was evaporated. The nature of this material is unknown; (*b*) granules of 100–150 Å diameter appeared at high magnifications which often seemed to be associated with a plastic material; (*c*) the existence of a thin membrane which may have originated from slightly soluble pectic substances or hemicelluloses. The results of these experiments are consistent with Ritter's findings that the native wood fibre is a capsular body with a central opening, the walls of which are made up of several sleeves which can be separated. The outer sleeves consist of fibrils wound tangentially and are probably destroyed during pulping, forming the fibral debris observed. The inner layers consist of fibrils running longitudinally which are telescoped into long tubular fibre sections during pulping and finally collapse into ribbons during the heating process. An attempt to correlate the performance quality of the paper with structural features was not successful.

Article by D. A. McLean H. A. Birdsall & C. J. Calbick *Industr. Engng. Chem.* 45, July 53 1509-1515

Sommaire : On a fait des recherches sur la structure des condensateurs au papier en employant la technique d'ombrage, méthode qui est décrite en détail, et en examinant les spécimens a l'aide d'un microscope électronique.

The Efficiency of Fluorescence in Cathode-Ray Tubes

210/I

Holland. The efficiency of fluorescent screens such as used in cathode-ray tubes has been investigated, *i.e.* the efficiency of converting the electron energy applied into luminous energy emitted from the screen. The screen normally consists of several layers of crystals of a size of 1 to several tens of microns each. An electron hitting a crystal will either be reflected (elastic collision) without any loss of energy or, more frequently, give up its energy to electrons in the crystal and these secondary electrons in the phosphor will pass their extra energy to luminescence centres and cause the emission of light. In order to obtain reasonable data electron acceleration potentials from 10 to 50 kV must be used which ensures the penetration of electrons to a depth of 25 μ in the crystals. To avoid that electrons pass through the screen without causing the emission of any light the initial measurements were taken on a phosphor layer, 0.3 mm. thick. A layer of this kind emits light to the back of the screen only. The light emission from a thick screen obeys Lambert's law. Thus, directing a beam of electrons of known current density to the screen at a certain angle and measuring the flux emitted at 90° yields the total energy radiated per unit area if multiplied with the factor π. The measured efficiency of η_m is then given by dividing this value by the product of current density and acceleration voltage. Making allowance for absorption of light within the layer the intrinsic efficiency is expressed by :—

$$\eta_i = \frac{2}{1 + R_\infty} \eta_m$$

where R_∞ represents the reflectivity of an infinitely thick layer. Details of the measuring equipment are given. An electron gun of the type used in electron microscopes emitted the electron beam through a hollow anode onto a metal plate mounted at an angle of 45° which had a number of recesses filled with the specimen materials. The plate was positioned in a cylinder fitted with various observation windows and could be rotated, permitting the measurement of the light emitted at various angles including that of 90°. The usual focusing arrangement was provided. The radiation emitted was measured in the range of 2,000–7,500 Å by means of a thermopile and in later measurements by means of photoelectric cells calibrated against the thermopile. In most experiments the beam had a density of 1μA/cm² and the acceleration voltage was 20 kV. In a special arrangement the effect of the temperature of the phosphor on the efficiency was investigated. The results of the experiments are given in a table partly reproduced on the next page. The values in column (1) represent the measured efficiency, (2) the aforementioned reflectivity factor, (3) the intrinsic efficiency, (4) the equivalent luminosity of the fluorescence calculated from the spectral distribution and (5) the luminous flux emitted per watt of primary electrons. The effect of temperature on the efficiency is shown in graphs. Normally, above a certain temperature, the efficiency is reduced. In the case of thin transparent fluorescent layers the luminous flux T emitted from the front of the layer and the flux R emitted from the rear are related as follows : $R + T < I_0$, where $I_0 = \eta_i I_p$, I_p being the intensity of the primary electrons. A graph is given showing the emission from the front and the rear as a function of the thickness of the thin layer. At optimum layer thickness the emission from the front does not exceed 40% of the total radiated energy. Losses due to incomplete coating vary from 5–15%. This condition can be improved by coating the back of the layer with an aluminium film; details of the procedure are given. The optimum layer thickness for the aluminium film is 0.02–0.08μ for 10 kV and about 0.1μ for 20 kV. At this thickness the reflectivity of the aluminium layer is 80% and the radiation from the front is improved by a factor of 2–2.2. The intrinsic luminous efficiency of a white-fluorescent sulphide mixture is about 60 lm/W. Thus, taking all losses into

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Efficiency of the Fluorescence of a Number of Phosphors under Electronic Bombardment at 20kV

Phosphor	ηm %	R_∞ %	ηi %	$\frac{Lm}{W_R}$	$\frac{Lm}{W_E}$	*Colour*
	1	2	3	4	5	6
ZnS–10^{-4} Ag–Cl	21			37	7	blue
(0.50Zn, 0.50 Cd) S–5×10^{-5} Ag–Cl	19.5			500	98	yellow
ZnO–Zn	7	85	7.5	340	24	green
$CaWO_4$	3	80	3.5	70	2	blue
CaO.MgO.SiO_2–0.03Ti	7	91	7.5	70	5	blue
Zn_2SiO_4–0.004Mn	8.5	99	8.5	475	40	green
$Zn_3(PO_4)_2$–0.3Mn	5	100	5	170	8.5	red
2CaO.Al_2O_3.SiO_2–0.04Ce	4	90	4			blue

account, the radiation intensity of the screen at the front can be expected to be 15 lm/W without aluminium backing and 30 lm/W with aluminium backing as long as the acceleration voltage is at least 10 kV.

Sommaire : Une discussion est faite sur le mécanisme de la luminescence cathodique et une description est donnée de la méthode permettant d'examiner la fluorescence des écrans de télévision.

Article by A. Bril & H. A. Klasens, *Philips Tech. Rev.* 15, Aug. 53 63-72

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An Inspection Method for Luminescent Screens

211/I

United Kingdom. A low pressure high voltage gas discharge within a cathode ray tube will excite luminescence in the screen. This phenomenon has been investigated as a possible basis for a method of screen inspection.

Sommaire : Des détails sont donnés sur une méthode practique d'inspection des écrans luminescents de téléviseurs, méthode qui peut être combinée avec le procédé d'aluminisation.

Letter by K. C. Burn *Vacuum* 3, Oct. 1953 415-417

Review of Work on Dichroic Mirrors and Their Light-Dividing Characteristics

212/I

United States. The light-dividing action of dichroic mirrors is based on the effect of interference in reflected and transmitted light. In order to achieve the desired action a glass plate is coated with a number of interference films each varying in thickness from $\frac{1}{4}$ of the wavelength of the light to $\frac{3}{4}$ of the wavelength of the light. The films are deposited by evaporation in a vacuum. First the action of a single thin film is considered. The interference effect of a $\frac{1}{4}$ wavelength film depends on the value of the refractive index of the material used. In a diagram it is shown that a film of a refractive index higher than that of glass will tend to increase reflection and diminish transmission. A film with a refractive index less than that of glass will tend to diminish reflection and increase transmission. If it is desired to obtain a higher reflection for a specified wavelength than is available from a single film of a given refractive index, a composite film is used consisting of several layers of alternatingly high and low-index values. The thickness of each is controlled so that the initial reflected rays at the specified wavelength are in phase when they leave the multiple film. The reflection obtained in this manner is then equal to the square of the sum of the amplitudes of the reflected rays. The reflection-transmission characteristics of a typical seven-layer film of this kind, giving a maximum of 90% reflection for light of 6,350Å at an angle of incidence of 45°, is shown in a graph. The light-dividing characteristics are governed by the angle of incidence as a change of the latter causes a change of path difference. Therefore, during the deposition process the glass supporting the film is positioned at an angle to the plane of the evaporator and a wedge-shaped deposit is produced. In use, the thicker part of the film receives the light incident at the greatest angle. A diagram is shown of the optical system of an RCA colour television camera where use is made of interference mirrors to divide the light into primary colours and to combine the primary colours for monitoring purposes. The films discussed so far are made from non-absorbing materials. However, light-absorbing materials can be used with advantage in transmission filters for the removal of blue light and the transmission of red and green light. By depositing first a relatively thin layer of low-index material and subsequently a comparatively thick layer of high-index material a film can be produced which reflects fairly evenly the light of all wavelengths of the visible spectrum up to 45%. Finally the design of heat-transmitting mirrors is discussed which reflect virtually all light of the visible spectrum.

Sommaire : Un article sur les derniers connaissances ayant trait à la fabrication des miroirs dichroiques et à leur pouvoir séparateur.

Paper by M. E. Widdop *J.S.M.P.T.E.* 60, April 53 357-366

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15 — PHYSICS — 15

The Importance of High Vacua to High Energy Nuclear Physics 213/I

See Abstract No. : 73/IV

The Velocity of Sound in Air at Low Pressures 214/I

Australia. Abbey and Barlow investigating the speed of sound travelling in a tube found an increase in sound velocity of 3.5 m/sec. when the pressure was reduced from 15 to 0.5 cm. Hg. Similarly Maulard found an increase of 4% in the pressure range 15–4 cm.Hg. The authors report on fresh experiments carried out in the pressure range of 760–5 mm. Hg. The experimental equipment consisted of a tube with a loud speaker at each end and two microphones attached to the tube walls, 4 m. apart from each other and each 3 m. away from the nearest loud speaker. The method consisted in measuring the travelling time of a sinusoidal train of waves, emitted by one of the loud speakers, between the two microphones. A diagram is shown of the experimental arrangement. Each experiment consisted of two runs, the signal being transmitted first by one loud speaker and then by the other. The result recorded represented the average time. Two types of tubes and two different signals were used : A bright steel tube of 5.07 cm. diameter, in which a 1,000 c/s sine wave was produced, and a tin-dipped steel tube of 19.05 cm. diameter, in which a 250 c/s sine wave was produced. The tube was pumped down to a pressure of 10^{-5} mm. Hg and contained carbon dioxide-free dry air. During one experiment the pressure never changed by more than 0.02%. In the 1,000 c/s operation the signal transmitted consisted of 17 cycles and in the 250 c/s operation it consisted of 5 cycles. The signals received by the microphones were amplified and applied to the Y_2 deflector plates of a double-beam oscillograph. By using a superimposed signal as a timing marker and an expanded time base (writing speed 8 cm. msec.$^{-1}$) and photographing the cathode ray tube display the time interval could be measured accurately to 1μ sec. in the experiments with 1,000 c/s and to 4μ sec. in the experiments with 250 c/s. The air pressure was measured with a U–tube manometer down to 5 cm. Hg and with a McLeod gauge at pressures below that. In the experiments with 1,000 c/s the temperatures were 20.7–23.1°C and in the 250 c/s experiments the temperatures were 21.6°C–25.5°C. The results of the measurements are given in two tables, one of which is partly reproduced below.

1000 c/s Results

P	$V_T(20°)$	A	B	C	D	E	$V(20°)$
760	342.84	0	0	0.002	0.624	–0.004	343.46
331	342.40	0.056	0	0.001	0.846	–0.010	343 29
199	342.10	0.073	0.001	0	1.220	–0.016	343.38
100.5	341.52	0.084	0.003	0	1.716	–0.032	343.29
50.6	340.88	0.092	0.007	0	2.420	–0.064	343.34
31.6	340.33	0.094	0.013	0	3.061	–0.101	343.40
20.8	339.74	0.096	0.022	0	3.770	–0.152	343.48
10.9	338.42	0.096	0.042	0	5.224	–0.286	343.50
7.81	337.28	0.097	0.057	0	6.157	–0.391	343.20
4.80	336.15	0.097	0.087	0	7.854	–0.623	343.57

Mean $W = 343.40 \pm 0.01$

The columns A–E give the values for the various corrections applied : A represents the corrections for gas imperfection which was carried out on the lines discussed by Hardy, Telfair and Pielemeier, B the corrections for attenuation based on an equation by Henry, C corrections for yielding of the tube wall, based on the findings by Land, D the tube corrections resulting from the accommodation coefficient corrections and E corrections for slip between the gas and tube wall. The authors applied a small correction to the temperature variable quantities in the Helmholtz-Kirchhoff equation, using for β_1 an experimental value equal to $(90.7 \pm 2.7)\%$ and $(93.5 \pm 17)\%$ of the theoretical value respectively. These values agreed with those of Kaye *et al.* established at atmospheric pressure. The experiments prove that the velocity of sound in free space is independent of the pressure down to 5 mm. Hg and the authors state that although the use of the values for β_1 may be open to argument the results established were not appreciably affected by it.

Sommaire : On donne une description détaillée des recherches effectuées afin de vérifier l'assertion que la vitesse du son dans des tubes augments pour des pressions bas mais on n'a pas remarqué que la vitesse du son augmente pour des pressions aussi faibles que 5 mm. Hg.

Article by D. E. Caro & L. H. Martin *Proc. Phys. Soc.* **66B**, Sept. 1953 760-768

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Has the Physics of Very Low Temperatures any Significance in Practical Work ? 215/I

See Abstract No. : 63/IV

On the Problem of New Methods of Heat Insulation of Very Low Temperature Apparatus 216/I

See Abstract No. : 183/III

Refractive Index and Thickness of Thin Aluminium Oxide Layers Produced Anodically 217/I

Germany. Two methods are described for the determination of the thickness and the refractive index of aluminium oxide layers. The layers were produced by the process developed by K. Strohmaier. One method originally described by G. C. Moench uses a Koesters interference prism and a mercury arc lamp as light source. If the oxide layer is placed in the path of the light the optical path is lengthened and a band shift produced which is related to the refractive index n and the thickness d by

$$Z_1 = (n-1)\frac{2d}{\lambda} \qquad (1)$$

In the second method the oxide layer, supported by glass, is covered with a coating of bismuth evaporated in vacuum. As a result a metal mirror is obtained with a small step on the border of the layer. Interference band shifts observed in this method are related to thickness by

$$Z_2 = \frac{2d}{\lambda} \qquad (2)$$

In both cases the band shifts observed were plotted as a function of the potential used in producing the oxide layer. Equation 2 facilitates determination of the thickness of the oxide film and proves that the thickness does not increase linearly with the potential. Dividing the values of the band shifts obtained by the first method, by those obtained by the second method yields the value of the refractive index. This value was found to be a constant, $n = 1.535 \pm 0.005$. The results of these methods are compared with the results obtained by Weiskirchner.

Sommaire : Description est faite de deux méthodes de mesure de l'épaisseur et de l'indice de réfraction des couches minces d'oxyde d'aluminium formées par procédé anodique.

Article by R. Bahn & O. Boettger *Z. Phys.* **135**, 1953 5376-5379

Improvements i.o.r.t. the Reduction of Reflection from the Surface of an Article 218/I

See Abstract No. : 145/III

High-Vacuum Evaporation 219/I

See Abstract No. : 136/III

On the Structure of Thin Silver Layers 220/I

Germany. Following up previous investigations of aluminium-silver alloys a comparison is made between electron diffraction micrographs obtained from the alloy and from pure silver respectively. It is shown that there is no hexagonal silver phase in thin evaporated layers of the alloy. The effects produced by the wobbling of the lattice and other lattice irregularities on the pattern shown in the micrographs have been studied.

Sommaire : On a fait l'étude de la structure cristalline des films composés d'un alliage d'aluminium et d'argent, et des films d'argent pur.

Article by H. Goettsche *Z. Phys.* **134**, 1953 517-525

The Effect of the Substrate Temperature on the Condensation Coefficient of Evaporated Antimony, Gold and Silver 221/I

France. After a brief reference to previous work on the effect of the thickness of the deposit on the condensation coefficient of antimony and cadmium evaporated onto supports maintained at normal temperature, the author reports the results of a recent investigation into the effect of the change in substrate temperature on the condensation coefficient of antimony and gold. These experiments were conducted with radio-active isotopes and confirm the theoretical assumption that the condensation coefficient decreases as the substrate temperature increases. In the case of antimony the decrease is considerable, even if the substrate temperature has been raised only slightly. In the case of gold the condensation coefficient hardly decreases at all and remains at a value near unity as long as the substrate temperature is not higher than 100°C. But when the temperature rises beyond 300°C the coefficient of gold decreases appreciably as long as the thickness of the deposit is small. The condensation coefficient depends not only on the thickness of the film but also on the nature of the film support as was to be expected from the results of the previous experiments with antimony and cadmium at normal substrate temperature. Experiments with silver indicate a value near unity as long as the film is thin and deposited on substrates maintained at normal temperature. Here, as in the case of gold, the condensation

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coefficient appears to decrease when the substrate temperature rises beyond 300°C. However, as the radioactivity of silver is poor no precise numerical values can be given.

Sommaire : Des expériences ont été faites avec trois métaux sur l'effet de la temperature sur le coéfficient de condensation.

Article by F. M. Devienne *Vacuum* 3, Oct. 1953 392-397

Metallic Grating Replicas as Internal Standards for Calibrating Electron Microscopes 222/I

See Abstract No. : 150/III

Narrow Pass Band Optical Filters 223/I

United Kingdom. A pass band filter is designed to transmit radiation between certain wavelength limits. Narrow pass band filters can be divided into three groups: Interference filters, polarisation filters and Christiansen filters. The first group consists of two partially reflecting metal films separated by a spacer film of dielectric material. This type of filter may be used in transmission or reflection. The second group depends on interference phenomena in polarised light and the third on selected scattering of the light by irregular particles suspended in a liquid. The article deals essentially with interference filters of the first group. The theory of the mechanism of filtration is discussed. The material normally used for filters operating in the visible spectrum is silver and the dielectric material for the spacer film is cryolite, magnesium fluoride or zinc sulphide. The thickness of the spacer layer determines the number of narrow pass bands in the spectrum. If there is only one pass band in the visible spectrum, the filter is called a first-order interference filter. The reflectivity of the silver films used is normally 90% and the absorption 5%. Absorption should be kept low in order to maintain high transmission. Reducing the reflectivity in order to obtain high transmission results in larger band width and a decrease between maximum and minimum transmission. It is concluded that the reflectivity must be 90% or over, if half-widths of less than 200Å are to be obtained. The filters are produced by thermal evaporation in vacuum onto glass as the substrate. Details of the procedure are given. The operational pressure is approximately 10^{-5} mm. Hg. All layers are deposited in one operational cycle without breaking the vacuum. The two important variables to be controlled are : The reflectivity of the metal films and the thickness of the dielectric film. The thickness of the first silver film is determined by observing the intensity of the filament through the deposit. The thickness of the dielectric film is determined by observing the colour of light reflected from a monitor plate which is covered up during the deposition of the first film. In the case of cryolite being the dielectric material the light reflected from a 60W bulb should be greenish-white if the film thickness required is 2,500 Å. The thickness of the second silver film is determined by observation of the filament intensity through the second half of the monitor plate hitherto unexposed. Greenland and Billington have developed a method for the precise location of the transmitted band, whereby polarised white light is directed at 45° onto the back surface of the filter and the reflected light focused on the slit of a grating spectrometer. The complete interference filter produces an absorption band which is complementary to the transmitted wavelength, λ_t. Prior to the deposition of the second metal film the combination produces an absorption band corresponding to a wavelength, λ_a. These two values have the relationship $\lambda_a = a\lambda_t + b$, where a and b are constants. Plotting λ_a against λ_t facilitates the construction of filters of any desired transmission within ± 20Å. Normally the transmission of a narrow interference filter is about 25%. This figure can only be improved if the silver layers are replaced by multilayer dielectric films. Details are given of an interference filter consisting of alternate dielectric films of high and low refractive index (four zinc sulphide layers and three cryolite layers) which have a peak transmission of 80% in the range of 4,500 to 6,500 Å and a half-width of 65Å. In the infra-red and ultra-violet range silver is unsatisfactory and aluminium has been used in a modified form. A suitable dielectric for that range is magnesium fluoride. About 100% transmission can be obtained in an interference filter of the frustrated total reflection type. It consists of a magnesium fluoride film deposited on the hypotenuse face of a 60° prism of high-index glass, a spacer layer of zinc sulphide and a second layer of magnesium chloride joined to a second prism by means of cement, the refractive index of which is the same as that of the glass. The article concludes with a short description of the special features of polarisation and Christiansen filters.

Sommaire. On décrit comment s'opère la construction et quelles sont les propriétés des filtres optiques ayant une bande de transmission très étroite.

Article by D. T. Turnbull *Lab. Practice* 2, Sept. 1953 480-487

An Inspection Method for Luminescent Screens 224/I

See Abstract No. : 211/I

The Efficiency of Fluorescence in Cathode-Ray Tubes 225/I

See Abstract No. : 210/1

Thin Layers Transmitting Short Ultra-Violet Rays 226/I

France. Transparent materials which could serve as support being sufficiently strong mechanically and heat-resistant or alternatively to be used as windows, not affected by ionic bombardment and vacuum-tight at the same time, are of great importance for the work in the field of spectrography below 1,200Å. The authors

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investigated the properties of certain metals such as beryllium and aluminium from this point of view. First they prepared silicon monoxide films which are known to have suitable properties by two methods specially developed for the purpose. In one the silicon monoxide film (evaporated from a mixture of silicon and silicon dioxide powder) is deposited on collodion film supported by a glass plate. The combined collodion-silicon monoxide film is removed from the glass by immersion in water and finally the collodion is dissolved in amyl acetate. In the other, the collodion film is formed on a specimen screen, the silicon monoxide evaporated onto it and the collodion dissolved subsequently. This latter method reduces handling of the film to a minimum. The transparency of the films $>10\ \mu g/cm^2$ produced in this manner is shown in a graph in terms of the extinction coefficient ($\log_{10} I_0/I$ in the range of 100–1,200Å. The value of the extinction coefficient of the combination (screen and film)-is fairly constant=0.55. As that of the screen is 0.35, the film itself has an extinction coefficient of approximately 0.2. The gas tightness of these films has not been investigated. The transparency of aluminium films prepared in a similar manner and backed permanently by the collodion coating rapidly deteriorates beyond 400Å. Gas leakage across a film with an area of about 0.2 cm^2 did not exceed 2×10^{-6} g/s when a pressure difference of 1 mm. Hg was applied across the film. Preliminary experiments with beryllium films gave similar results.

Letter by N. Astoin & M. B. Vodar *J. Phys. Radium* 14, June 1953 424-425

Sommaire : On a étudié différents matériaux qui pourraient être employés comme supports ou fenêtres sous form de couches minces, en fonction de leur résistance au bombardment ionique et de leur transparence optique.

Photoelectric Cells for the Vacuum Ultra-Violet 227/I

United States. Due to the lack of suitable window material there are no photo-electric cells readily available which could be operated in the ultra-violet at wave-lengths below the transmission limit of quartz. Radiation at wavelengths shorter than that can only be detected indirectly by means of luminescence or directly by the use of windowless photo-tubes. This article is only concerned with the latter. The performance of windowless phototubes is governed by two major factors, the quantum efficiency which is normally low in that range and the photo-electric properties of the metal surface used. Kenty and Baker were the first to investigate yields of several metals in the short wavelengths region quantitatively. The former did not give detailed distribution curves of yield *vs.* wavelength and the latter used lithium fluoride covered cells and was therefore limited to the wavelength of 1,050Å. The authors investigated nickel, tungsten and platinum and obtained yields down to a wavelength of 850 Å and in addition yields at the helium resonance radiation wavelength of 584 Å. The results are shown in a graph. Due to the gap in the range measured the maximum yield for each metal investigated could not be properly determined. The graph shows yields as high as 10 photo-electrons per 100 incident quanta for nickel and tungsten near 1,000 Å. Details of the experimental arrangement are given. The pressure in the phototube was approximately 10^{-5} mm. Hg, *i.e.* the same as that prevailing in the chamber of the monochromator used and the reproducibility of the values obtained was independent of pressure in the region below 0.5 micron Hg. Repeated exposure to air did not affect the performance. This latter result of the experiments is especially important as there is no windowless secondary electron multiplier commercially available yet which operates reproducibly at 10^{-5} mm. Hg or after exposure to air. In conclusion the significance of the results of the authors' experiments is discussed with regard to the development of photocells with lithium fluoride windows and photon counters.

Article by H. E. Hinteregger & K. Watanabe *J. Opt. Soc. Amer.* 43, July 1953 604-608

Sommaire : La construction et les performances de cellules photo-électriques spécialement developpés pour les mesures de rayons ultra-violet de très courts longeurs d'onde.

Methods and Techniques for the Determination of Specific Surface by Gas Adsorption 228/I

See Abstract No. : 64/IV

Fundamental Aspects of Combustion—Some Reactions of Atomic Hydrogen in Flames 229/I

See Abstract No. : 234/I

A High Temperature Radiation Calorimeter 230/I

United States. A spherically symmetrical calorimeter operated in a high vacuum has been developed for making direct measurements at high temperatures. The instrument is suitable for the study of solid phase transformations and heat capacities of metastable phases facilitating an experimental procedure superior to indirect methods. The calorimeter proper is a sphere machined from high-purity graphite rod. Its diameter is about two inches. The sphere has a blind hole which was threaded and could be closed with a graphite plug which carries the internal heater. The latter consists of a thin lava cylinder supporting a winding of 28 (B. and S.) Nichrome wire. The centre of the cylinder was filled with another graphite plug which made good contact with the calorimeter at one end and the larger plug at the other end. The calorimeter was positioned in the centre of a spherical shield furnace. The shield furnace consisted of a ceramic outer shell, a graphite liner and a thin ($\frac{1}{4}$ inch) layer of plastic alundum cement in order to obtain a truly isothermal surface surrounding the calorimeter. The furnace carried a Nichrome winding embedded in the graphite layer. For temperature measurement one thermal element was fitted to the calorimeter and a second to the shield furnace in appropriate positions. As it was observed that some graphite evaporated during operation, specially designed ' film breakers ' were used to keep the calorimeter electrically isolated from the graphite shield liner. An all-metal vacuum system was employed served by an oil diffusion pump. The operational pressure was

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0.01 micron Hg at room temperature. Operating at 600°C the pressure could be maintained at 0.1 micron Hg. The vacuum chamber was immersed in a water tank. Brief observations are given on practical difficulties encountered in operating the vacuum system. The temperature measurements were carried out with platinum / platinum–13% rhodium thermo-elements. Details of the circuitry required to control the internal heater and the shield heater are given. The theory of the instrument is presented for the following conditions: The rate of heat transfer from the surface of the sphere to the surface of the shield, cooling characteristics of a perfectly conducting sphere, and those of a sphere with a finite thermal conductivity, cooling characteristics of the sphere, when heated internally by a non-leaking heat source and alternatively when heated internally by a leaking heat source, cooling characteristics of the sphere at varying shield temperatures. Calibration data and cooling curve data were obtained experimentally. Calibration data was obtained for the two cases (*a*) the heat input to the shield was constant and (*b*) the temperature of the shield furnace was constant. The data obtained for constant shield temperature gave straight lines, the heat transfer constant increasing linearly with the cube of the average temperature. Similarly, cooling data was obtained for both cases. In an experiment conducted at 663°K the heat capacity of the calorimeter was determined to be 41.7 cal. deg.$^{-1}$ and since there were 9.435 moles of graphite this represented an atomic heat capacity of graphite of 4.42 cal. deg.$^{-1}$ mole^{-1}. The respective values shown in the literature are 4.26 and 4.29.

Report by I. Johnson *Oklahoma Agr. & Mech. Coll. Research Rep.* Proj. No. NR-358-269 August 1953

Sommaire: Un calorimètre sphérique qui fonctionne sous vide a été construit. La théorie de l'instrument est expliquée et les resultats des expériences, faites pour vérifier la théorie, sont discutés.

Apparatus for Low Temperature Calorimetry

231/I

United Kingdom. Two apparatus are described which have been designed for the measurement of the thermal properties such as specific heat of solids over a continuous range of temperature with a bottom limit of 2°K. Liquid helium is used in both cases for obtaining the low temperatures required. One apparatus is of the cryostat type and employs liquid helium supplied from a separate liquefier, the other, an expansion apparatus, embodies a liquefier of the Simon type. The actual calorimeter containing the specimen under investigation is suspended in each case in an evacuated enclosure, the walls of which have to be cooled down to the required temperature. In the cryostat the calorimeter is fully enclosed by a radiation shield which is joined to a copper vessel used as the helium reservoir. The helium is introduced into the vessel by syphoning. The evaporating helium is returned to the laboratory gas holder by a separate tube. Details of the syphoning system are given. The cryostat has a capacity of 100 c.c. About an equal amount of liquid helium evaporates during the transfer. At a satisfactory vacuum in the enclosure about 10 c.c./h of liquid helium is evaporated. The essential component of the expansion apparatus is the expansion chamber, a copper ' bomb ' of about 100 c.c. capacity, into which helium gas at about 100 atmospheres pressure is admitted. Fitted to the top of the ' bomb ' is a vessel which is filled with liquid hydrogen when a temperature of 20°K has been reached during the initial operation of cooling down the apparatus with liquid oxygen. Subsequently, pumping gently, some of the hydrogen is evaporated, as a result of which the remaining hydrogen solidifies in a few minutes. At this stage the lowest obtainable temperature has been reached and the helium in the ' bomb ' is expanded back to the gas holder. The design of the calorimeter is the same for both apparatus. It consists of a drum with a removable lid and is fitted with vanes in order to promote rapid establishment of thermal equilibrium in the specimen. Its dimensions are: diameter –45 mm. and height –45 mm. The wall thickness is 0.2 mm. The heat capacity of the calorimeter and that of several substances investigated in these apparatus are given in a table partly reproduced below.

Heat Capacities of Calorimeter and Typical Specimens

°K	*Calorimeter*	*Cu*	$CaCO_3$	*Polythene*
2	0.001	0.003		
10	0.024	0.097		0.10
40	0.62	6.2	3.31	1.56
100	2.31	26.8	12.7	4.30
300	3.92	41.0	26.6	14.7

The thermal unit is calories . deg^{-1} throughout.

Providing there are facilities for storing liquid helium it takes no longer than 1½ hours to obtain liquid helium in the cryostat, the corresponding period for the expansion apparatus is 2–4 hours. The expansion apparatus has proved more flexible than the cryostat, whereas the cryostat allows for speedier operation than the expansion apparatus.

Article by R. W. Hill *J. Sci. Instrum* 30, Sept 1953 331-334

Sommaire. On donne la description de deux modèles de calorimètres travaillant à basse température. Dans les deux modèles on emploie de l'helium liquide; dans l'un un liquefier à helium du type Simon est incorpore, dans l'autre l'instrument comporte un appareil pour l'emploi avec un liquefier séparé.

Two New Forms of Melting Point Calorimeters

232/I

United States. Two designs of melting point calorimeters are described which were developed for the purpose of determining the purity of gaseous hydrides of boron in preparation of experimental low temperature work on these materials. The first was suitable only for the measurement of melting point as a function of fraction

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melted but the second incorporated facilities for the measurement of the heat of fusion. The samples required for a determination did not exceed 4 and 3 ml. respectively, and the time taken for a determination was 2 hours in the case of the first design and 3 hours in the case of the second. The calorimeter proper in the first design consisted of a glass tube 35 cm. long and 12 mm. diameter, sealed at the bottom end. Inserted into it was a platinum resistance thermometer element, a coiled wire, which was used as the heater at the same time. The calorimeter tube was inserted in a glass tube of 40 mm. diameter which formed a jacket and could be evacuated to 10^{-5} mm. Hg. In operation the sample was introduced into the calorimeter tube to a depth just covering the thermometer junction and the whole assembly was placed in a suitable refrigerant bath. After the temperature of the sample had been reduced to approximately 20°C below its melting point the jacket was evacuated, and the experiment was started. Results obtained on three samples of diborane are given in a table reproduced below

Purity Determination on Diborane

Sample	*Determination*	*Tm, Ohms*	*Purity, %*
A	1	63.9568	99.950
A	2	63.9572	99.938
B	3	63.9564	99.968
C	4	63.9265	99.30

Tm Melting Point of Sample

The sample C was prepared from B by adding 0.59 mole % of ethane as an impurity. The second design represented a simple form of a vacuum calorimeter made from a copper-to-glass Housekeeper seal, closed at one end. The resistance thermometer heater was wound round the outer surface of the copper portion of the seal and was in intimate contact with a copper block attached to the glass tube about 0.5 inch above the glass-to-copper seal. The copper block was provided with a heater which could be very accurately controlled. The whole assembly was surrounded by a vacuum jacket and during operation the copper block was heated to the melting point of the sample. The sample was melted completely during a run which permitted determination of heat capacities and heat of fusion. Results are given of a test run with pure n–pentane and the same material carrying iso–octane as an impurity.

Article by J. T. Clarke H. L. Johnston & W. De Sorbo *Analyt. Chem.* 25, August 1953 1156-1159

Sommaire : On donne la description de deux nouveaux modèles de calorimètre à point de fusion.

A Vacuum Microsublimation Apparatus 233/I

See Abstract No. : 241/I

16 — CHEMISTRY — 16

Fundamental Aspects of Combustion—Some Reactions of Atomic Hydrogen in Flames 234/I

United Kingdom. Many chemical reactions are known to involve the participation of atoms and free radicals. A flame is a chemical reaction emitting light. By examining the spectrum of the light emitted the presence of certain species can be proved and by burning flames at reduced pressures (4–10 mm. Hg), these measurements can be rendered more informative still. Of particular interest in combustion reactions is the behaviour of hydrogen and oxygen atoms. This review is essentially concerned with the detection of atomic hydrogen in flames. Reference is made to previous research work on the hydrogen spectrum by Balmer and on the properties of atomic hydrogen by R. W. Wood and Bonhoeffer. The recombination of hydrogen atoms is an exothermic process and the heat of reaction is approximately 102 kcal. Recombination is promoted by the presence of a third body either in the form of a surface or of gaseous molecules. After discussing the mechanism of slow combustion of hydrogen and carbon monoxide as far as it is known today the authors turn to the conditions applicable in burning solid fuel which depend critically on the presence of elementary particles such as atomic hydrogen, etc. The presence of atomic hydrogen in flames can be detected by means of chemiluminescence produced by certain flames if brought into contact with promoted oxide surfaces. The radiation obtained is in excess of that to be expected according to the laws of radiation and has been established to be due to an impurity (activator) present in the parent lattice of the luminescent material. Oxides of calcium, strontium, barium and beryllium containing bismuth, manganese, lead and antimony as activators give a strong coloured radiation when in contact with a hydrogen flame. Details of an experiment are reported, where atomic hydrogen was produced in a discharge tube, 1 inch in diameter and 46 inch. long, filled with H_2. A side tube joined to the centre of the discharge tube served as a connection to a ratory pump of a speed of 2 ft.^{3}/min. The latter tube was fitted with a conical joint, through which specimens of activated oxides could be introduced. The discharge was run at 20 mA and the pressure was 1–5 mm. Hg. The colour of the discharge served as an indication whether atomic hydrogen was produced in adequate quantities. On heating the support carrying the specimens the luminescent colour obtained approached that observed in the flame proving that recombination of hydrogen atoms took place on the oxide surfaces. In a special series of experiments the combustible gas was led through a block of refractory brick and burned on the top face of of the block which carried a thin layer of the activated

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oxide. With hydrogen the characteristic luminescence appeared in the form of an annulus on the surface of the brick. Attempts were made at reducing or suppressing the chemi-luminescence by adding permanent gases to the hydrogen burner. It was found that with the exception of argon, which is an atomic gas, adequate concentration of the additive gases could extinguish the chemiluminescence. This phenomenon is attributed to separate reactions which remove hydrogen atoms and while there is no definite knowledge on the nature of these reactions it can be assumed that the added gas acts as a third body promoting the recombination of hydrogen atoms. A table is shown giving the critical concentration of additives required to suppress the chemi-luminescence. More work is called for in order to establish a comprehensive theory of flame propogation in particular on the question of the part played by radical concentrations present in the reaction zones of flames.

Sommaire : Un mémoire est présenté ayant trait aux différents techniques employées pour la detection de la présence d'hydrogène et d'oxygène atomaire dans les flammes.

Article by J. R. Arthur & D. T. A. Townend *J. Inst. Fuel* **26**, July 1953 44-51, 61

Methods and Techniques for the Determination of Specific Surface by Gas Adsorption 235/I

See Abstract No. : 64/IV

Metal Column for Distillation of Corrosive Gas Mixtures at Low Temperatures 236/I

United States. Details are given of a fractionating column which yields pure fractions or at least samples of a mixture of a comparatively small number of components of complex gaseous mixtures containing corrosive materials such as fluorine, chlorine or hydrogen fluoride. There are a number of low temperature distillation columns available but none is suitable for operation with corrosive mixtures. The design described is largely based on a construction described by Podbielniak. The construction materials were nickel, bronze and chlorotrifluoro-ethylene polymer. The column was a 30-inch nickel tube, 5/32 inch in diameter, packed with 3/32 inch helices. The distillate was condensed at the top of the column which could be cooled with liquid nitrogen. At the lower end was a still pot of 20 ml. capacity, heated electrically. The column was surrounded by a jacket which could be evacuated or serve as a passageway for the coolant. Details of the instrumentation and of the automatic coolant supply system are given. In operation a sample in vapour form was admitted to the evacuated still pot and cooled by liquid hydrogen. The still pot was then heated until the desired pressure was obtained. This took about 10–20 minutes. After recording temperature and pressure in the column the distillate was withdrawn into an evacuated product receiver of known dimensions and the pressure in the receiver was determined. The results of a test run are given in a table partly reproduced below.

Results of Fractionations of Freons

Run No.	*Compounds Charged*	*Boiling Point °C*	*Gas Charged C.c.*	*Maximum Take-Of Rate C.c./Min.*	*Break Point*	*Purity of Plateau Material %*
1	Freon 12 Freon 114	–29.4 + 3.0	441 452	20	441 c.c. Freon 12	99 + 99 +

The normal withdrawal rate in this experiment was 20 c.c. per minute but was reduced to 5 c.c. per minute, when the temperature of the distillate rose. The purities were determined by the gas density method. Due to the thermal conductivity of the metals used in the construction the precision of the results obtained was lower than that of other analytical low temperature gas distillation apparatus.

Sommaire : On fait la description d'une colonne de distillation permettant de séparer, à basse température, des mélanges très hetérogènes de gaz corrosifs.

Article by N. C. Orrick & J. D. Gibson *Analyt. Chem.* **25**, July 1953 1100-1103

Vapour Pressures of Pyridine and Its Homologues 237/I

United Kingdom. Accurate vapour pressure data for pyridine and its homologues (α–picoline, β–picoline, γ–picoline, 2 : 5–lutidine and 2 : 6–lutidine) is of technological importance in connection with the separation of these materials from mixtures such as tar, shale and petroleum fractions by means of fractional distillation. The article reports on a method (and its results) for the determination of the vapour pressures of the above materials in the temperature range from 50°C to the normal boiling point. The boiling points of the liquid were measured by a dynamic method and the apparatus used was a modification of that described by Willingham *et al.* A duplicate boiler was added for observing the boiling point of a purified sample of water prepared by the technique of Emeléus *et al.* under the same pressure conditions. The two boilers were connected to a common exhaust line through isolating cold traps. Details of the arrangements are given in a sketch. The pressure was controlled by a coarse and a fine manostat. Details of the latter, which operated within exceptionally fine limits, are given. The vapour pressures were calculated from the temperatures of the vapour of boiling pure water using the data compiled by Osborne and Meyers. All measurements were carried out in a nitrogen atmosphere. The performance of the apparatus was checked in test runs with pure benzene. A table is given showing the values obtained in the experiments indicating at the same time the amounts by which they differ from those calculated on the basis of published data.

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Pyridine

Purity 99.85 ± 0.007 moles %

P	*t*	$\triangle t$
764.89	115.473	−0.003
493.23	100.994	0.000
63.80	47.327	−0.020

α-Picoline

Purity 99.70 ± 0.15 moles %

P	*t*	$\triangle t$
773.28	130.037	+0.010
491.90	114.552	−0.008
79.96	64.363	−0.006

β-Picoline

Purity 99.71 ± 0.14 moles %

779.98	145.101	−0.005
501.21	129.368	0.000
96.54	81.282	−0.008

γ-Picoline

Purity 99.75 ± 0.12 moles %

762.00	145.462	+0.008
490.98	129.837	+0.001
77.62	76.906	−0.004

2 : 5-*Lutidine*

Purity 99.85 ± 0.07 moles %

765.90	157.299	−0.003
495.78	141.497	+0.002
71.76	85.098	−0.034

2 : 6-*Lutidine*

Purity 99.63 0.03 moles %

765.22	144.300	+0.003
497.55	129.190	+0.002
87.06	79 290	−0.016

P - mm mercury at 0°C and standard gravity, t-C

$\triangle$t obs.temp. - calc. temp.

Sommaire : On a fait un rapport sur une méthode permettant de determiner la tension de vapour de la pyridine et des matériaux associés. Des résultats obtenus par cette méthode sont aussi consignés dans ce rapport.

Article by E. F. G. Herington & J. F. Martin *Trans. Faraday Soc.* **49**, Feb. 1953 154-162

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Additive for Jet Combustion Fuel 238/I

See Abstract No. : 187/III

A Replica Technique in the Study of Chemical Precipitation Processes 239/I

See Abstract No. : 157/II

The Determination of Moisture in Fat 240/I

Russia. Methods of drying and determining the moisture content of edible and non-edible fats are discussed. Non-edible fats after drying by ordinary heat treatment in a chamber at approximately 100°C showed a moisture content of up to 3 times as much as that of material dried under vacuum. Edible fats, however, showed a moisture content 7–10 times less after normal heat treatment than after drying in vacuum. A table is given of the moisture content of a wide range of edible fats, determined (*a*) after normal chamber drying at about 100°C and (*b*) after vacuum drying at 70–75°C. Results vary appreciably. This is attributed to the instability of the materials in question, which are known to oxidise at temperatures above 80°C. A simple apparatus for the vacuum drying of fat is described. The operational pressure in the chamber was approximately 100 mm. Hg produced by a water jet pump. During operation carbon dioxide gas was passed through the chamber continuously. The pressure was read on a dial gauge and the heating was effected by placing the drying chamber in a hot-water bath.

Sommaire : On décrit les expériences qui ont été faites en vue de déterminer la quantité d'humidité contenue dans les graisses comestibles et non-comestibles.

Article by A. Yefimova *Meat Industr.* (Russian) **24**, No. 5, 1953 54-56

A Vacuum Microsublimation Apparatus 241/I

United States. A simple vacuum sublimation apparatus is described which facilitates the isolation of micro-quantities of biological substances in extremely pure form from small pieces of tissue or small quantities of fluid. The top of a Pyrex test tube, 30 × 200 mm., is ground to give a vacuum-tight fit with the aluminium block of a Fisher-Johns melting point apparatus. The material to be processed, in a microbeaker fabricated from test tubes 50 mm. wide, is placed on the aluminium block and the Pyrex tube is passed over it to form a hood. A vacuum is drawn through a side tube joined to the hood and the metal block is heated. After completion of the drying process the vacuum is broken, the hood removed and a microscope cover slip placed over the mouth of the beaker. The hood is reassembled, vacuum applied and the block heated to the temperature required in order to effect sublimation. Using this apparatus 4 μg amytal have been recovered from 2.5 ml. of whole blood.

Sommaire : On donne la description d'un appareil d'opérer la sublimation sous vide pour isoler facilement des quantités très petites de substances biologiques.

Note by J. R. Maher *Science* **117**, 15.5. 1953 529-530

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The Frozen Bone Bank of the Netherlands Red Cross 242/I

See Abstract No. 68/IV

Freeze Drying and the Tissue Bank 243/I

United Kingdom. The clinical aspects of tissue transplantation are discussed. Non-vital methods of tissue storage to facilitate transplantation are considered. After a short survey of atmospheric methods the author deals extensively with the merits of the freeze drying technique and gives details of a practical method of freeze drying arteries for the purpose.

Sommaire : L'aspect medical de la transplantation de tissus est discuté on fonction des méthodes practiques de lyophilisation des artères.

Article by H. H. G. Eastcott *Vacuum* 3, July 1953 279-286

Comparison of the Survivals of Aspergillus Spores Lyophilised by Various Methods 244/I

See Abstract No. : 71/IV

Preservation of the Virus of Tomato Spotted Wilt in Dried Plant Material 245/I

See Abstract No. : 70/IV

Thin Layers Transmitting Short Ultra-Violet Rays 246/I

See Abstract No. : 226/I

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Crystallographic Variations of Field Emission From Tungsten 247/I

See Abstract No. : 79/IV

Electron Currents in Thin Oxide Films on Aluminium 248/I

United Kingdom. Aluminium anodised in suitable electrolytes formed thin insulating oxide films which acted as the dielectric of a condenser. The formation process involved a flow of ion currents and this was studied in a previous paper. At voltages below the formation voltage the leakage current through the oxide film was an electron current, i_-. The relationship between this current, the field F across the film and the temperature T was investigated. Assuming electron flow to be inhibited by a potential barrier the current was shown to follow the theoretical equations $i_- = A_- \sinh B_- F$, where A_- and B_- were constants depending on T and the barrier dimensions opposing the current. The theoretical equation was verified by comparison with a number of experimental determinations of the current-voltage and current-temperature curves. At very low fields hysteresis effects could be observed when the voltage was changed making reproducible results difficult. This effect was ascribed to space charge formation within the oxide layer due to trapping of electrons and the possible mechanism of this was discussed. At intermediate values of the field excellent agreement between predicted and experimentally determined curves was obtained. At high fields the increase in current above that predicted was ascribed to the onset of ion current. From results for both, aluminium and tantalum, the constants A_- and B_- were obtained and hence values for the theoretical potential barrier and temperature dependence calculated. By taking these results together with those previously obtained for the ion current, the total current for the complete current-voltage curve at various temperatures could be investigated and close agreement of theory with typical measurements are shown.

Sommaire : Pendant l'electrolyse d'une surface d'aluminium, une fine couche d'oxyde isolante est formée par l'action des ions à la tension appliquée. La nature et l'effet du courant de fuite constitué par des electrons à d'une tension basse par rapport à la normale ont été étudiés.

Article by A. Charlesby *Proc. Phys. Soc.* **66B**, July 1953 535-541

Refractive Index and Thickness of Thin Aluminium Oxide Layers Produced Anodically 249/I

See Abstract No. : 217/I

An Electron-Micrograph Study of Oxide Films on Electropolished Surfaces of Iron 250/I

Canada. Electropolishing is known to give a very high degree of polish on the metal surface, higher than mechanical polishing. The result of certain experimental work seems to indicate that the electropolished surface is clean and not covered by an oxide film. However, more recently evidence has been found that

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electropolished metal surfaces are covered by very thin films. The authors examined electropolished surfaces of iron of a composition C–0.005, Si–0.0075, S–0.013, P–0.003, Mn–0.007, Ni–0.018, Cr–0.02, Cu–0.003, 0–0.12, N- 0.008 and H–0.00009 % which was treated in a bath, consisting of 850 c.c. ethyl alcohol, 70 c.c. water, 50 c.c. concentrated perchloric acid and 23 c.c. concentrated hydrochloric acid, in an electron microscope. The bath had a temperature of 18°C and a current density of 1 A/cm² was used. Treatment lasted 30 seconds. The changes in surface structure produced by oxidation and reduction were used to establish the significant features of the electropolished surface and chromium shadow-cast replicas of the specimen surface were made by a method explained in detail. In order to obtain information as accurate as possible the oxidations and reductions were carried out in an infra-red furnace described previously (*Can. J. Technol.* 30, (1952), 63). The surfaces investigated were either electropolished only or reduced after electropolishing or oxidised after electropolishing or, finally, reduced and reoxidised after electropolishing. Results are shown in reproductions of the electron micrographs. Comparison with a mechanically polished surface confirms that the electropolished surface is relatively smooth. Reduction with hydrogen results in a furrowed surface which is broken in certain places indicating that the film on the surface is most probably an oxide film. The thickness of this film is estimated to be about 100 Å. The film is built up during the electropolishing and drying process but it cannot be said for certain whether it is originated during the former or during the latter.

Article by E. J. Caule & M. Cohen *Canad. J. Chem.* 31, March 1953 207-211

Sommaire : On donne la description des techniques employées pour faciliter l'observation au microscope électronique de l'état de surface du fer poli par procèdé électrique.

Determination of Hydrogen in Wrought Aluminium Alloys

251/1

United States. In order to avoid porosity in aluminium welds the hydrogen content of the material must be as low as possible. In this connection it is necessary to know the amount of occluded hydrogen as distinct from that held by hydrated surface oxide. Most of the existing methods of hydrogen determination are either not sufficiently accurate in this respect or not sufficiently rapid when applied to commercial wrought aluminium alloys. The article reports on the procedure and results of a modified tin fusion method which is satisfactory and facilitates the determination of hydrogen contents of 0.1 c.c./100 g. The materials examined were the following commercially available alloys : 2 S, 24 S, 43 S, 61 S. 74 S and 56 S. Details of their composition are given. The tin had a purity of 99.8 %. The apparatus consisted of a furnace section, a collecting section and a gas analysis section. The furnace section consisted of a vertical borosilicate glass tube holding 200 g. of tin surrounded by a wire-wound furnace. A horizontal tube joined to the vertical tube was used for storage and pre-heating. A liquid nitrogen cold trap was added to freeze out the moisture. The collecting section consisted essentially of a continuously pumped fixed calibrated volume where the total quantity of gas evolved from the material was measured with a McLeod gauge. From here the gas was transferred with the help of a Toepler pump to the analytical section where the hydrogen was removed from the system by diffusion through a heated palladium tube. Details of the procedure are given. In order to determine the correct amount and distribution of the hydrogen in a particular alloy three analyses have to be carried out : (*a*) An abraded sample of the material ' as received ' was analysed. This yielded the total hydrogen content, (*b*) An abraded example was pre-heated for 4 hours at 500°C in vacuum and subsequently analysed without re-abrading or exposure to the atmosphere. This yielded the value of the residual internal hydrogen after the surface hydrogen and nearly all of the internal hydrogen had been removed, and (*c*) a sample was pre-heated as before but subsequently re-abraded and exposed to the atmosphere before it was analysed. During the exposure to air the same amount of surface hydrogen as present in the first sample was re-absorbed as hydrated oxide. A table, partly reproduced below gives the results obtained and an indication of the distribution of the hydrogen in the materials tested.

Distribution of Hydrogen in Wrought Aluminium Alloys

Volume of Hydrogen per 100 *g. in c.c.*

Type of Alloy	*A* *Total (as Received)*	*B* *Residual internal (preheated)*	*C* *Residual internal plus external (preheated) reabraded*	*D* *Internal removed by preheat A–C*	*E* *Total internal D + B*	*F* *External C–B*
2S	0.5	<0 1	0 5	<0.1	<0.1	0.5
43S	1.0	0.3	0.6	0.4	0.7	0.3
61S	0.8	0.1	0.6	0.2	0.2	0.6

It is shown that the hydrated surface oxide yields appreciable quantities of hydrogen. Experimental welds of 43S wire on 61 S baseplate were analysed and showed an internal hydrogen content of at least 1.2 c.c. 100 g. In theory this amount of hydrogen should produce about 10% porosity and this was confirmed by radiographic examination to be the case in practice.

Article by C. B. Griffith & M. W. Mallett *Analyt. Chem.* 25, July 1953 1085-1087

Sommaire : On donne la description d'une nouvelle méthode permettant de détecter l'hydrogène contenu dans les métaux légers.

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Improved Process for the Preparation of Metallic Powders

252/I

France. It is a known fact in powder metallurgy that the fineness of the metallic powder produced is the greater, the lower the temperature at which reduction is carried out. Extreme fineness of powder is required for the production of high-quality tungsten carbide tools, for instance. It has been observed, however, that the powder produced is pyrophoric if reduction takes place below a certain temperature. This applies in particular to metals of the iron group. A new method is described which is claimed to eliminate the pyrophoric properties of fine metallic powder. Two alternative forms of treatment are suggested. The first provides for the cooled metal powder to be transferred to a separate chamber which is subsequently evacuated. Before the material is removed from that chamber nitrogen or carbonic acid gas is introduced. The second consists essentially of cooling the powdered metal gradually to a temperature below zero in an evacuated chamber. Tungsten, for instance, reduced from WO_3 by hydrogen at about 720°C looses its pyrophoric properties if it is cooled down to, and kept at –20°C for a period ranging from 30 minutes to 2 hours. Tungsten carbide produced from tungsten treated in this manner has a Rockwell hardness of A 93 compared with A 91.8 obtained in the conventional process.

Regie Nationale Des Usines Renault *Brit. Pat.* 670,900

Sommaire : Au sujet de la préparation d'une poudre métallique, des détails sont donnés sur une méthode de réduction qui facilite le traitement à assez basse température éliminant les propriétés pyrophoriques de la poudre.

Process for the Purification of Titanium

253/I

See Abstract No. : 169/III

The New Metal Titanium

254/I

United Kingdom. Titanium has been known to exist for more than 150 years, but its production in appreciable quantities is only 5 years old. Van Arkel and de Boer were the first to produce pure titanium in 1925. Currently the bulk of titanium is produced by the Kroll process. The output of titanium in the U.S.A. in 1953 was estimated to be 10,000 tons. Details of the industrial Van Arkel and Kroll processes are given. Alternative methods of extraction, all still in the experimental stage, are mentioned, such as reducing the tetrachloride with sodium and a two-stage reduction process of the oxide. Experiments were also conducted with an electrolytical method using fused lithium or potassium chloride as electrolytes but the deposited metal could not be satisfactorily separated from the electrolyte. Melting of the metal as much as the extraction has to be carried out in the absence of air. In addition, as titanium attacks all the refractory materials normally used in metal melting, it must be carried out by methods not involving their use. An arc-melting furnace, originally developed by Bolton for tantalum in 1905, is described in detail and shown in a sketch. It allows for the production of ingots of up to 4,000 lb. Titanium undergoes an allotropic transformation at 882°C. Above this temperature it has a body-centred cubic structure (β-phase) and below this temperature it has a hexagonal close-packed structure (α-phase). The latter phase has a low c/a ratio (1.587), *i.e.* the dominant deformation mechanism is slip on the prismatic and not on the basal planes. High-purity titanium has an ultimate tensile strength of about 15 tons/sq. inch. Commercially pure titanium has an ultimate tensile strength of about 35 to 40 tons/sq inch. with 30 to 25 % elongation. The oxygen content of commercial titanium is 0.25 % and the nitrogen content is 0.1 %. Increase of the oxygen content makes the metal brittle, but with certain reservations hot-working operations can be carried out in air at temperatures not exceeding 1000°C. The maximum permission carbon content is 0.5%. Cold-working operations are hampered by the tendency of the metal to seize on other metals, when in sliding contact under pressure. Remelting of scrap is not possible because of the lack of suitable arc furnaces and of the cumulative contamination which results from repeated melting and fabrication. Further details are given with regard to elastic modulus, fatigue properties, impact strength and creep properties. The two most outstanding properties of the material are its strength : weight ratio and its corrosion resistance. The role played by the various known alloying elements is not fully explored yet. Iron, chromium, manganese,

Table II. Approximate Compositions and Properties of α/β Titanium Alloys

Alloy Type	*Nominal Alloy Content, %*						*Condition*	*Typical Mechanical Properties*		
	Cr	*Fe*	O_2	*Mo*	*Mn*	*Al*		0.2% *P.S.* tons/ inch.²	*U.T.S.* tons/ inch.²	*Elongation*, %
Titanium-chromium-iron	1.8 2.7 3.0	0.9 1.3 1.5	0.15 0.25 0.5	— — —	— — —	— — —	Hot-worked and annealed at 700°C	35–45 55–65 65–75	50–60 65–75 75–85	15–25 10–20 5–15
Titanium-chromium-iron-molybdenum	2.0 4.5	2.0 4.5	0.2 0.2	2.0 4.5	— —	— —	Hot-worked and annealed	50–60 55–65	60–70 65–75	10–20 5–15
Titanium-manganese	—	—	—	—	7.0	—	Annealed sheet	60–65	65–70	10–20
Titanium-manganese-aluminium	—	—	—	—	4.0	4.0	Hot-worked bar	60–65	65–70	15–25
Titanium-chromium-aluminium	5.0	—	—	—	—	3.0	Hot-worked bar	60–75	70–85	5–10

molybdenum, vanadium, tantalum and niobium depress the transformation temperature and are more soluble in the β-phase than in the α-phase. Aluminium and tin, for instance, have the opposite effect. Alloys available and some of their properties are shown in a table, reproduced on previous the page. None of the alloys shown can be welded. It is hoped that weldable alloys will result from research work now being conducted on single phase alloys.

Sommaire : Un article général sur les propétés de titanium et les dernières méthodes de fabrication de ce métal.

Paper by M. Cook *J. Inst. Metals* **82**, 1953-54 93-106

Technique of Melting and Casting under High Vacuum

255/I

Germany. As early as 30 years ago the production of alloys by melting in vacuum was started on an industrial scale but full development of this technique was delayed until large-capacity diffusion pumps could be made available for the purpose. For large-scale operations pumps with a speed of 500 to 2,000 l/s in the pressure range of 10^{-3} to 10^{-5} mm. Hg are required, but in view of the large quantities of gas to be handled by the pumps good speeds are also required in the range of 10^{-1}—10^{-3} mm. Hg. If possible, the vacuum chamber should be fabricated from stainless steel which has a smooth surface, facilitates cleaning, and reduces the amount of gases liberated during evacuation to a minimum. The crucibles should be made from a refractory material of great density. In order to keep the evolution of gas low, different materials have to be chosen for the crucible to suit the different materials to be processed. Two principally different methods of heating are employed, resistance heating and induction heating, but only the latter is of practical importance in industrial operations. The constant agitation of the liquid metal produced by this form of heating constitutes a decided advantage as it facilitates the liberation of gases in the melt. In addition, it permits a rapid increase in temperature of the melt which is important in the production of alloys where the temperature of the melt has to be reduced every time a new constituent is added. Comparatively volatile constituents should be added to the melt as late as possible, *i.e.* immediately prior to casting, in order to avoid loss by evaporation. The vapours rising from the bath tend to cover the observation window in the furnace with an opaque deposit. Methods of counteracting this effect are discussed. The presence of the metal vapours may result in an electrical discharge and for that reason the induction coil should not be operated at a voltage higher than 250, when a frequency of 10 kHz is used. The basic features of various casting arrangements are described, a.o. the bottom pouring device providing for a plug of the same material as the processed material, which is kept cool initially and finally removed by melting. The mould should preferably be of steel or cast iron which will be heated up slightly by the stray field of the induction coil and thus evolve little or no gas during the casting process. Mention is also made of centrifugal casting in vacuum. The author gives details of various production procedures and certain processing times. For instance, a plant of 4 litres capacity should require 3–5 minutes for pumping down to the operational pressure of 10^{-3} mm. Hg. In the case of the processing of steel alloys very violent gas liberation occurs in the initial stages and therefore the pump has to be shut off until the metal is fully liquefied. During that period carbon oxide gas is admitted which acts as a blanket. When the liberation of gases is completed pumping is resumed. A charge of 25 kg. will require approximately 1 hour processing time. The vacuum immediately prior to casting is approximately 10^{-3} mm. Hg. In conclusion the author lists the advantages of vacuum melting and by way of example gives a table, partly reproduced below showing the effects of melting under high vacuum in the case of Siemens-Martin-Steel.

The Effect of Remelting of Steel Under High Vacuum on the Composition of Steel

Material	*Charge kg*	*State*	*Chemical Composition*							*Total*	*Nitrogen*	
			C %	*Si* %	*Mn* %	*P* %	*S* %	*Cr* %	*O* %		*Dissolved*	*Not Dissolved*
Siemens-Martin-Steel	Appr. 1.5	Before	1.01	0.33	0.31	0.027	0.010	1.64	0.0023	0.0067	0.0042	0.0025
		After	0.96	0.33	0.03	0.029	0.010	1.58	0.0001	0.0025	0.0019	0.0006

Sommaire : Toutes les méthodes connues de fonte et moulage sous vide sont examinées, et les caractéristiques de construction des pompes employés pour des fours à vide sont données.

Article by C. Winkler *Stahl & Eisen* **73**, Sept. 1953 1261-1267

Vacuum Melting Lengthens Fatigue Life, Improves Impact Properties

256/I

United States. This is a short report on the outcome of comparative fatigue tests of alloyed steel type 52100, which is widely used in the manufacture of bearings, produced by the conventional method in one case and by vacuum melting in the other. The exact composition of both varieties of steel are given in tabulated form. The commercial material was in the form of a ¾ inch-diameter rod, in a spheroidised condition. The vacuum-treated material was reduced to the same diameter from 3 inches by hot-working and was then normalised (by heat treatment), air-cooled and spheroidised. All subsequent machining to prepare specimens for the tests were identical for both types of material. Fatigue testing was carried out in a Krouse rotating beam machine. The results obtained are shown in a graph, where stress (psi) is plotted against frequency of stress load application. The limiting values were not established but, at low stresses, the vacuum cast-material gave values generally two orders of magnitude greater than those of the conventional material. A 50% overall improvement was recorded at the same life.

Sommaire : On enumère les avantages que donne l'application des procédés de fonte sous vide à un acier de composition spéciale.

Article by J. H. Moore *Iron Age* 2.4.1953 154-156

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Induction-Melting Furnaces

257/I

Germany. In the case of induction-heated furnaces the energy required to produce the heat is withdrawn as electrical energy from a coil in the primary circuit and induced in a secondary circuit, an electro-magnetic field acting as the link. The secondary circuit is set up in the material to be heated. The amount of heat generated follows Joule's law and depends on the current induced in the secondary circuit. The basic formulae for the transfer of energy from the primary to the secondary circuit are given. The value of the current induced depends on the number of windings of the primary coil and on an adequate intensity of the magnetic flux. The author considers the case of inducing current in a thin-walled piece of tubing situated inside of, and coaxial to, the primary induction coil, and derives the basic formulas for the magnetic flux, induced voltage and induced current applicable in that case. He shows the change in the distribution of the magnetic flux density if a second tube of smaller diameter is introduced, but the same laws do not apply if a solid body is substituted for the two tubes. Therefore in order to facilitate proper calculations he introduces the term of 'penetration depth' (of the magnetic flux in the solid body), which is given as

$$\delta = 50.3 \sqrt{\frac{\varrho}{\mu f}} \qquad \text{(cm)}$$

where ϱ is the specific resistivity, μ is the permeability of the material and f the frequency employed. Sufficient energy is absorbed by the solid body if its dimensions are $d \geqq 3.5\,\delta$ (d for diameter). Similar formulae are given for the electrical efficiency of the induction heating system and for the determination of the frequency required. The magnetic field exerts a mechanical force. This force is the sum total of an electrical, a magnetic and radiative force and produces persistent agitation of the melt in the crucible, resulting in the liquid forming a raised cap in the centre of the bath. This phenomenon determines the position of the coil surrounding the crucible and the dimensions of the crucible, in particular the ratio of diameter to depth. There are various designs of induction furnaces. In some cases the energy is induced in the charge itself, in others it is induced in the crucible. Metallic crucibles have the advantage of facilitating the melting of non-conducting materials but should not be used if there is a danger of alloying with the crucible material. Ideal materials for crucibles are ceramics. The cheapest form of induction furnace is that employing the frequency of the supply. Medium frequencies, *i.e.*, 50–10,000 c.p.s. are normally employed where ceramic crucibles are used and can be produced by rotary generators. High frequencies are generated by electronic apparatus and are normally used for special processes only. Typical vacuum induction furnaces employ frequencies up to 10,000 c.p.s. and low primary voltages say 300V in order to avoid arcing due to the metal vapours present in operation.

Article by K. H. Brokmeier *Glas & Hochvakuum Tech.* **2**, Sept. 1953 234-240

Sommaire : L'équipement électrique des fours à induction fait l'objet d'une discussion. Cette discussion porte sur la théorie et les modèles de les fours.

The Properties of Cast Chromium Alloys at Elevated Temperatures. I. The Melting and Casting of Chromium-Rich Alloys

258/I

See Abstract No. : 166/III

Vacuum Die-Casting

259/I

United States. In die-casting machines where the die cavity is evacuated prior to each casting operation the die is normally fitted with a rubber gasket on the peripheral face of one of the die sections for the purpose of obtaining a vacuum-tight seal. In operation these gaskets are exposed to an excessive squeezing action and to the heat developed in operation of the machine which soon results in permanent deformation of the gasket thus destroying the effectiveness of the seal. Both these causes of failure of the seal are eliminated in an arrangement proposed in this patent. The rubber gasket is fitted to the outer perimeter of the cavity in the movable die in such a manner that it surrounds the cavity completely and extends radially inwards. The fixed die is tapered and its upper perimeter is small enough to permit the gasket to pass over it without touching when the movable die is advanced into the closing position. As the latter moves further downwards the gasket makes sliding contact with the tapered surface of the fixed die, until in the fully closed position the inner edge of the gasket snaps into a groove provided on the fixed die. A vacuum-tight seal is effected automatically as soon as the cavities in the die are evacuated in the conventional manner prior to casting. As the seal is in a position, a considerable distance away from the molten metal and, as the two die sections are water-cooled, the material of the gasket is protected from the heat developed in the process.

R. F. Plott & U.S. Atomic Energy Commission *U.S. Pat* 2,637,882

Sommaire : Des détails sont donnés sur une machine de moulage à moule métallique, où le moule est evacué avant chaque opération de moulage.

Improvements r.t. the Metallic Lubrication of Ball Bearings

260/I

See Abstract No. : 169/II

19 — SCIENCES (other than the above) — 19

Metallising of Glass, Ceramic and Plastic Surfaces

261/I

See Abstract No. : 137/III

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II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

Ultimate Pressure Obtained by Oil Diffusion Pump 129/II

Japan. An equation for the ultimate pressure P of an oil diffusion pump is derived from a modified Gaede principle. This equation is examined by experiments with di-octyl hexahydrophthalate and silicone oil DC 702. In the lower range of cooling temperature T_1, P decreases with the increase of operating temperature T_2 and this leads to an increase of the sealing effect of the jet. In moderate T_1, P remains constant and is in equilibrium with the vapour pressure of the oil film near the ionisation gauge. In the higher T_1 range, P increases with the decrease of the intrinsic pumping speed for exhausting the gases liberated from the wall or produced by the decomposition of oil vapour in the gauge. A minute amount of impurities in the oil greatly affects P.

(*Chemical Abstracts*)

Article by H. Ishii *Oyo Butsuri* **22**, 1953, 69-72

Sommaire: A l'aide d'expériences pratiques on a opèré la vérification d'une équation permettant de déterminer la pression minimum que permet d'obtenir une pompe à diffusion à huile. Cette équation est derivée du principe de Gaede modifié.

Improvements in High Vacuum Diffusion Pumps 130/II

United States. Diffusion pumps of the fractionating type consist of a cylindrical vertical casing which holds a number of concentrically arranged vapour chimneys fitted with jet nozzles at the top. At the lower end the chimneys rest on the bottom plate of the casing, sub-dividing the boiler into an equal number of concentric boiler sections. These boiler sections are connected with each other by small apertures at the lower end of the chimneys to permit the pump fluid to flow from one section to another. In order to ensure proper fractionation of the oil during its travel and to make sure that oil of the lowest vapour pressure component only is rising up the chimney fitted with the jets nearest to the high vacuum inlet of the pump, the oil should flow on a tortuous path and the flow should be restricted to the path established by the apertures in the chimney. In conventional designs it is difficult to avoid general seepage of oil from one compartment to the other for reasons connected with the use and construction of the pump. If seepage was to be avoided, the chimneys should be welded to the bottom of the casing which causes difficulties in cleaning the pump, or, alternatively, baffles should be used welded to the bottom plate of pump which increases the cost of manufacturing the pumps. Exact alignment between the bottom of the pump casing and the bottom of the chimneys is difficult to obtain and in operation can be easily impaired by slight warpage of both components. It has been proposed therefore, to assemble the chimneys separately as a sub-assembly and to add to the lower ends of each chimney a collar of a diameter slightly smaller (or larger, as the case may be), than that of the respective chimney in order to avoid contact with the chimney, and to fit the collars in such a manner that they can have a limited vertical movement with respect to the chimney. This sub-assembly can be placed on the bottom plate of the pump casing without forming an integral part of it. In the case of faulty alignment between the lower ends of the chimneys and the bottom plate of the casing, etc., the collars will drop into a position adjusting this condition and preventing the seepage of oil between two compartments in these areas. Higher ultimate vacua and increased pumping speeds have been obtained with pumps constructed in accordance with this design.

Eastman Kodak & M. E. Johnson *Brit. Pat.* 693,814

Sommaire: Description des améliorations d'une pompe à diffusion à huile qui empêchent le suintement pendant le fractionnement du fluide.

The Use of Silicone Oil as an Operating Fuel for Diffusion Pumps 131/II

Germany. A short report is given of the performance obtained with diffusion pumps operated with a silicone oil, the data of which are given in the table below.

Physical Data of the Silicon Diffusion Pump Fluid 4018/50 *D.*

Density of 20°C.	0.948
Mol. Weight	Appr. 1400
Viscosity (eSt)	
At 20°C.	17
At 100°C.	5.2
Flash Point (°C.)	Appr. 220
Specific Heat (Cal/g)	0.26
Cubic Expansion Coefficient	0.0010
Boiling Point (°C.)	
At 760 mm. Hg	Appr. 360
At 10^{-3} mm. Hg	Appr. 100
Vapour Pressure at 20°C. in mm.	10^{-7}

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The fluid was tested in two-stage glass diffusion pumps. The fore vacuum was approximately 10^{-3} mm. Hg. The ultimate vacuum obtained in the tests varied between 10^{-5} and 10^{-6} mm. Hg. These pressures could be obtained without difficulty even after repeated exposure of the oil to air. Execssive agitation of the oil and erratic boiling could be traced to excessive heating. The oil was also tested in 2– and 3–stage metal diffusion pumps and a pressure of 10^{-5} mm. Hg was invariably obtained. No changes in the oil could be observed even after an exposure to air lasting more than 100 hours, but it required a recovery period of some 5 minutes before normal pumping efficiency was obtained after exposure.

Sommaire: On a fait des recherches sur une huile de silicone de consistance donnée pour déterminer son aptitude à servir de fluide dans une pompe.

Article by E. L. Holland-Merten, H. Reuther & S. Sliwinski *Chem. Tech.* 5, June 1953 301-303

Experimental Investigations in Some High Vacuum Systems

132/II

Jugoslavia. Construction of Hickman-type oil diffusion pumps and of the gauges required for low and high vacua is described. With Apiezon oil, silicone fluid and Octoil (di–2–ethyl–hexylphtalate) as pumping fluids, pumping speeds were estimated to reach 10 l./sec. The vacua obtained were satisfactory (0.012 μ Hg) for work in electronics for which the pumps had been designed.

(*Science Abstracts*)

Sommaire: On écrit un rapport sur les résultats de certains expériences sur les systèmes à vide poussé equipés avec des pompes à diffusion du type Hickman.

Article by J. Loncar *Period. Math. Phys. Astron.* (*Zagreb*) 7, No. 4, 1952 197-215

Apparatus for Evacuating Hollow Articles

133/II

See Abstract No.: 173/II

An Automatic Toepler Pump and a Circulating Pump

134/II

United Kingdom. An automatic Toepler pump is described which embodies a sintered glass disc sealed in the glass and covered with a pool of mercury replacing the conventional non-return valve. The disc is capable of withstanding a pressure difference greater than one atmosphere. The ultimate pressure obtainable with the pump is about 0.1 mm. Hg. The second type of circulating pump uses again sintered glass discs as valving elements. The pumping speed of the latter is 0.5 l./hr. This pump is useful where the rate of purification of the mixture of gases is slow.

(*Author*)

Sommaire : Détails d'un projet spécial d'une pompe automatique Teopler et d'une pompe à circulation montée avec des disques de verres poreux à la place de valves ordinaires.

Letter by E. J. Wilson *Vacuum* 3, July 1953 287-288

Liquid Ring Pump

135/II

United States. The improved design of a liquid ring pump is described. The pumping action in a pump of this kind is effected by a ring of liquid which is driven by a rotor, the blades of which are open both ends and form so-called buckets of liquid. The chamber wall is formed to provide alternate lobes and lands. The lands form a surface which is cylindrical and coaxial with the rotor. The lobes increase in depth adjoining one land, *i.e.*, during the suction sweep, and decrease in depth in the area adjoining the following land, *i.e.*, during the discharge sweep of the bucket. During the suction sweep the liquid is forced out of the bucket by centrifugal forces drawing the gas into the inner end of the bucket through a connection to the vessel to be pumped and during the discharge sweep the liquid is forced back into the bucket causing the gas in the bucket to be compressed and discharged through an outlet port in the chamber wall. The lands are provided to separate these sweeps and in the case of a 20–blade rotor no work is being done by any bucket over 36° of each revolution. That represents a loss of about 10% of the available working space. Accordingly it is proposed to eliminate the lands in order to improve the capacity of the pump and in order to obtain lower pressures in the vessel to be evacuated. The upper and the lower lobe in the new design have a cylindrical surface with an axis parallel to that of the rotor but situated in a position offset from the rotor axis by the same amount each. As a result the liquid in the bucket crossing the area, where the 2 lobes join, will start its outward movement while the bucket is still connected to the outlet port but no loss in operatiog efficiency from this cause could be traced.

Sommaire : Détails des caracteristiques d'une pompe récente à anneau liquide prevu pour augmenter l'efficacité de ce type de pompe.

Nash Engineering & H. E. Adams *Brit. Pat.* 694,694

New Developments in the Production and Measurement of Ultra-High Vacuum

136/II

See Abstract No.: 191/I

Has the Physics of Very Low Temperatures any Significance in Practical Work ?

137/II

See Abstract No.: 63/IV

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21 — GAUGES — 21

Experimental Investigations on Some High Vacuum Systems 138/II

See Abstract No. : 132/II

New Developments in the Production and Measurement of Ultra High Vacuum 139/II

See Abstract No. : 191/I

Research on the Friction Manometer 140/II

Germany. The friction manometer originally proposed by Haber-Kerschbaum in 1914 for the measurement of pressures in the region 10^{-1} to 10^{-4} mm. Hg has been improved by Coolidge and finally, in 1940, by Wetterer. The latter joined 2 quartz fibres to a quartz strap, the assembly forming a triangle and carrying a small quartz cup at the point where the 2 quartz fibres were joined. This assembly is kept under mechanical tension and therefore oscillated in one plane only. The relation of the half-time value t_h to pressure p is given by

$$t_h = \frac{J.\ \ln 4}{p\ \sqrt{M}\ a + z}$$

where J is the momentum of inertia of the system, M the molecular weight of the gas, z the self-damping constant, and a another constant. In theory a system of this design should facilitate the measurement of pressures as low as 10^{-10} mm. Hg and the authors investigated the design details in order to extend its range as low as practicable. To increase the range of pressures to be measured the cup should be as large as possible and at the same time as light as possible so that the momentum of inertia of the system is kept at a minimum. The following are the most favourable dimensions which can be obtained in practice : Surface area 1.5 sq. cm, thickness of the cup material 3–5 μ, length of the quartz strap, forming the base of the triangle, 35 mm., height of the triangle 55 mm. The width of the quartz fibres suspended from the strap should be 15 μ. In theory pressures of 10^{-8}mm. Hg should be measurable with this system. The instrument was used experimentally for the determination of the vapour pressures of mercury. The results are shown in a graph, proving that the instrument gave reliable values down to 10^{-7} mm. Hg. Further experiments determining the vapour pressures of organic compounds, such as Apiezon greases, etc. showed that the instrument would yield only precise values where the molecular weight of the gases in question was known. In the case of the organic compounds mentioned the instrument could only be used to determine the order of magnitude of the prevailing pressure.

Article by H. Klumb & H. Kollmann-sperger *Glas & Hoch-vakuum Tech.* 2, August 1953 211-213

Sommaire : Pour l'étude du principe de travail d'un manomètre à fibre de quartz une serie d'expériences ont été faites.

A New Manometer for the Pressure Region 10^{-2} to 10^{-6} mm. Hg 141/II

Germany. The authors refer to a radiometer-type manometer which was developed by Klumb in 1944 for use in the pressure range of 10^{-2} to 10^{-7} mm. Hg, the operation of which was independent of the nature of the gaseous atmosphere. In the present article the authors report on investigations into certain design features of the gauge which aimed at a construction suitable for practical needs. In the finally developed form the measuring element consists of a cylinder with a multitude of vanes cut out of the cylinder surface and bent outwards. This cylinder is suspended under tension in a frame by a combination of tungsten or quartz fibres A small mirror is fitted to the torsion suspension. The heater element placed in a small glass cartridge is situated within the cylinder. Two bi-metal strips are attached to the outer face of the heater element in such a manner that they stop any movement of the cylinder until the heater is operated. Then the bi-metal strips contract and release the measuring system. This latter device and the new form of suspension make the instrument vibration-proof and safe in transport. The frame with the components described is sealed into a metal tube which carries a window at the level of the mirror on the suspension, in order to facilitate indication of the pressure reading on a scale fitted to the exterior of the tube by means of a projected light beam. The walls of the tube housing the instrument are water-cooled and are surrounded by a damping magnet. The glass panel forming the window is sealed into a cap formed from special sheet metal and the cap is soldered onto the tube, an arrangement which obviates the use of cement or other materials which may tend to degas. If housed in a metal tube, the instrument has a range from 10^{-2} to 5×10^{-6} mm. Hg. If housed in glass, the range extends to 10^{-7} mm. Hg. The methods of calibrating the instrument are discussed.

Article by H. Klumb & E. Weissmann *Glas & Hoch-vakuum Tech.* 2, Sept. 1953 266-269

Sommaire : Détails de réalisation et performances d'un manomètre du type radiomètre récemment developpè.

A Simple Diaphragm Micromanometer 142/II

United Kingdom. Mass spectrometers employed for the analysis of multi-component gas mixtures should preferably be fitted with means of measuring directly the pressure of the samples admitted. As the pressures to be measured range from 20 to 100 μ Hg, a 'mechanical' gauge of the Bourdon tube or diaphragm-type should be suitable. In the present article a simple diaphragm micromanometer developed for the purpose is described. The diaphragms used were about 0.001 inch thick and 3 inch. in diameter, flat in the centre over a width of 0.75

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inch diameter and corrugated over the remaining area of the surface. The corrugations were formed by a spinning process, described in detail, and were about 0.01 inch deep. Only copper and silver foil was found to be free from pin holes and porosity in this application. The finished-machined diaphragm had a turned down rim about 0.03 inch deep which after assembly of the diaphragm was soldered to the support in the gauge head. The upper condenser plate, 11/16th inch wide and mounted on a bridge, faces the flat centre part of the diaphragm at a distance of about 0.01 inch. A circuit diagram is given. Only one valve, an electron-coupled oscillator, is used. Any change in capacity is converted into a frequency change. The capacity change depends on the position of the diaphragm. If the diaphragm is in a neutral position, *i.e.* both sides are evacuated to the same pressure, the frequency is about 6.6 Mc/s. If the pressure difference across the diaphragm is about 100 μ Hg the frequency is decreased to about 5.8 Mc/s. The dial of the instrument has 100 equal divisions but as the response of the instrument is not linear, due to the particular type of electrical equipment used, a calibration curve is required. Two standard and one special method of calibration are mentioned. In the case of the latter a 5 l. gas reservoir and the manometer to be calibrated are joined to the inlet leak of the mass spectrometer. A sample of gas is filled into a 5 m l. gas pipette, its pressure in the pipette is determined and then the gas is transferred to the reservoir by means of a Toepler pump. The calculated pressure of the gas in the reservoir is compared with the respective reading of the micromanometer. In operation, at a pressure difference of 200 μ Hg the diaphragm touches the upper condenser plate. In this manner the diaphragm is protected against excessive movement which may result in permanent distortion.

Article by D. B. Cook & C. J. Danby *J. Sci. Instrum.* **30**, July 1953 238-240

O *Sommaire* : Construction simple d'un diaphragme micromanomètre pour l'emploi dans les spectromètres de masse afin d'analyser un mélange de gaz.

Sorption and Desorption of Gas in the Cold-Cathode Ionisation Gauge

143/II

United Kingdom. It is known that, as distinct from the case of the hot-cathode ionisation gauge, the cold-cathode Penning gauge sorbs gases in quantities which affect the readings for many gases and vapours. The experiments reported here form an extension of the work carried out by Penning (*Physica* **4**, 1937, 71). The gauge employed in the experiments had a cylindrical anode, 2 cm. diameter and 2½ cm. long, which was operated at earth potential. The end plates were approximately 0.3. cm. away from the anode and where at a potential variable from 0 to–4,000 V. The magnetic field had a strength of 900 G along the axis of the cylinder. The experimental arrangements incorporated a 3–inch. oil diffusion pump (Apiezon A oil), a needle valve and 2 hot–cathode ionisation gauges. One of the hot-cathode ionisation gauges was situated in front, and one, together with the Penning gauge, at the end of a U–shaped tubulation leading through a cold trap. Calibration curves of the Penning gauge for argon are given. The rate of gas clean–up was determined on the base of the formula

$$Q = C\ [(P_1 - P_1^1) - (P_2 - P_2^1)] \qquad (1u\ sec)$$

where C is the conductance of the piece of tubing between the gauges, P_1 and P_2 the pressure readings at either end of the tube with the Penning gauge working, and P_1^1 and P_2^1 the corresponding pressures with the Penning gauge switched off. The following gases were investigated : nitrogen, air, water vapour, ethyl ether, carbon tetrachloride, argon and hydrogen. Except ethyl ether all gases gave similar results which are shown in graphs. Ethyl ether was the only gas where sorption could be observed to continue for some time after the Penning gauge had been switched off. The results for permanent gases were independent of the use of the trap. The Penning gauge absorbs residual vapours coming from the oil diffusion pump in the same way as the hot cathode ionisation gauge. In an experiment with a high-speed ionisation gauge in front of the tubulation the ultimate pressure of the pump was indicated to be 2×10^{-5} mm. Hg on the ionisation gauge, and 10^{-7} mm. Hg on the Penning gauge. The desorption rate at room temperature never exceeds 1 % of the sorption rate observed, but can be speeded up by baking the gauge. The gas given up during bursts caused by baking is of the same order
O of magnitude as the gas originally sorbed. The error of reading of the pressure caused by the pumping effect of the discharge can be appreciable and is demonstrated for the case of nitrogen in a table. No definite conclusions can be drawn on the clean-up mechanism. The clean-up may result either from ions being driven into the gauge electrode or by gas adsorbed on electrode material sputtered onto the walls of the gauge envelope. The results of the experiments indicated that the former type of mechanism is more likely to be responsible for the clean-up but the latter type cannot be completely ignored in view of the results with ethyl ether.

Article by J. H. Leck *J. Sci. Instrum.* **30**, August 1953 271-274

Sommaire : La vitesse de l'abaissement de pression causé par la jauge à ionisation à cathode froide a été étudiée.

On the Conditions of the Performance of an Absolute Mercury Manometer

144/II

Russia. A description is given of the Pearson Manometer used for measuring pressures of gas mixtures containing up to 90% of water vapour. The range of pressures measured varied from 0.01 to 1.5 mm. Hg. The sensitivity over the entire range was 0.002 mm. The original paper by Pearson omitted several important considerations for effective use which are here described. These include the strict thermostating of the manometer below room temperature (15°C), the reduction of jolts and shakes, the purity of the mercury and the cleanliness of the glass surfaces. When the manometer is used for thermal analysis of gaseous mixtures it is necessary to take into account the temperature of the gas investigated and to correct by the Reynolds formula. The use of such a manometer is recommended for the analysis of gaseous mixtures containing easily condensable components.

(*Science Abstracts*)

Article by V. T. Slavianskii *Zh. Tekh. Fiz.* **22**, No. 11, 1952 1881-1884

Sommaire : Déscription d'un manomètre à mercure Pearson employé pour la mesure des pressions de mélanges de gaz contenant jusqu'à 90 % de vapeur d'eau.

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Abstract No. and References

22 — MEASURING PLANT (VACUUM) — 22

Crystallographic Variations of Field Emission From Tungsten 145/II

See Abstract No. : 79/IV

The Electron Telescope 146/II

France. The physics of the electron telescope are discussed in great detail. Formulae have been developed for the case where the objective consists of a plane cathode and 2 diaphragms which facilitate rapid calculation of the position of the plane of the image and the magnification obtained. The smaller the diameter of the diaphragms, the greater is the concentration of the electron beam. The value of the magnification obtained depends on the ratio of the field strength in front and behind the first diaphragm. A method of calculation is proposed for the determination of the path of electrons in the marginal regions, whereby the direction and speed of the electrons can be precisely defined. Applying this method to the conditions in an electron telescope with a curved cathode it is shown that distortion of the image is produced which cannot be counteracted by adjusting the curvature of the cathode surface. An improvement of image definition can only be obtained by increasing the potential fall in the region surrounding the cathode. Ions originating from the ionisation of molecules of the residual gas in the tube do not seem to interfere with the clarity of the image but primary electrons impinging on the diaphragms give rise to secondary emission of low velocity electrons which intensify the stray light phenomena along the edges of the fluorescent screen. On the other hand, secondary electrons of high velocity, *i.e.*, reflected primary electrons, have no tangible effects. Finally, the importance and various causes of noise have been analysed and methodically investigated. (See Vol. I, Abstract No. 26/II).

Sommaire : La théorie et réalisation du telescope à electrons sont discutées.

Article by M. M. Duchesne *Bull. Astron.* 17, No. 1, 1953 pp. 88

Metallic Grating Replicas as Internal Standards for Calibrating Electron Microscopes 147/II

See Abstract No. : 150/III

Electron Diffraction 148/II

United Kingdom. Due to the small penetrating power of the electron, electron diffraction presents a suitable means for studying the surface structure of materials. Basic details of the theory are discussed. The diameter and appearance of the diffraction ring on the camera screen depends on the lattice spacing in the crystals of the material investigated and on the wavelength of the electrons. At an accelerating voltage of 50 kV the wavelength is about 5×10^{-8} cm. As the wavelength is dependent on the accelerating voltage, the latter has to be kept at a constant value. The gas pressure, the current passing in the discharge and the accelerating voltage are related to each other and in order to stabilise conditions in the camera the gas pressure is kept constant by means of a special leakage system and the supply and the discharge current are kept constant by a complex electronic circuit, a diagram of which is shown for the case of the Finch Camera. The lattice parameter of the diffraction pattern can be calculated with an accuracy of $\pm$ 2 % from the data of the apparatus and the value of the accelerating voltage but in the Finch camera provision is made to eliminate the need for such calculations by arranging for the comparison of the patterns obtained from the material investigated with 'standard' patterns of substances, the surface structure of which is known. The electron beam is collimated by a fixed diaphragm positioned close to the anode, and by an adjustable diaphragm. Finally, the beam is focused by a focusing coil on the fluorescent screen. Ions and stray electrons are trapped. There are separate diffusion pumps for the discharge and the camera chamber. Only a few minutes are needed to pump down to operational pressure. The electron camera is used for the investigation of corrosion phenomena, catalytic activities on surfaces of films, paper structure, quality of metal spraying and the performance of electrodeposition. When a metal is electrodeposited on a single-crystal surface under conditions favouring lateral growth it can be demonstrated by means of electron diffraction that the deposit forms a coherent extension to the single-crystal substrate. Where deposition takes place on a polycrystalline surface a thin deposit will give electron diffraction patterns identical to those of the substrate but when a certain thickness is reached the crystal orientation of the deposit differs from that of the substrate and is characteristic of the conditions of the bath only. Regions of misfit due to the mosaic of the substrate crystal would not coalesce during deposition. ' Slip ' resulting from deformation can be spotted by means of electron diffraction as the pattern produced shows the boundary changes due to screw and edge dislocation and the interaction of these dislocations.

Sommaire : Réalisation, performance at applications de la camera à diffraction d'electron avec références particulières sur la camera de Finch sont examinées.

Article by A. E. Williams *Mech. World & Engng. Rec.* 133, Sept. 1953 390-394

A New Electron Optical Bench 149/II

Switzerland. The main features of the improved design of an electron-optical bench are described. The image on the fluorescent screen can be measured directly, either photographically or by means of a microscope. During use the whole bench can be kept under observation. Two vacuum chambers can be accommodated, thus offering the opportunity of lengthening the optical axis. Three electron-optical elements can be placed on the bench and their position along the axis of the bench can be adjusted accurately without breaking the

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vacuum. One of these elements can be displaced with equal precision in a direction rectangular to that of the axis of the bench. The accelerating voltage employed can be twice as high as in the case of the conventional design. The operational vacuum is of the order of 10^{-5} mm. Hg. The single flat-shaped guide rail normally provided is replaced by 4 round driving shafts.

Sommaire : Un nouveau banc d'optique électronique est décrit.

Article by E. Bas *Helv. Phys. Acta* **26**, 15.6.1953 438-439

Polythene Windows for Vacuum Systems

See Abstract No. 182/III

150/II

Electrode Contamination in Electron Optical Systems

See Abstract No. : 196/I

151/II

Large Bent Crystal Vacuum X-Ray Spectrograph

152/II

Sweden. An instrument with high resolving power for small intensities is described, designed for use in high vacuum. The angular range covered is 35–80°, and the range of wavelength covered is 3,500–19,600 XU. (*B.S. I.R.A. Bulletin*)

Sommaire : Détails d'un spectrographe à rayons–X sous vide, à large crystal courbé.

Paper by A. E. Sandstroem *Ark. Fys.*, **4**, 1952 517-534

A Vacuum Microbalance for Measuring Sorption in Dielectrics

153/II

United Kingdom. For the measurement of the rate of sorption of water vapour by non-polar dielectrics a microbalance was required with a sensitivity of 1 in 10^6. The balance had to be suitable for operation in vacuum or at low pressures of a vapour and had to withstand sudden pressure changes. An instrument developed for the purpose is described in this article and is based on a design by Pettersson. The balance beam was rhombic in shape, constructed from 0.4 mm. hard-glass fibres with a tie bar along the short diagonal. The cross bar of 1 mm. diameter was joined to the centre of the tie bar. A mirror was fitted to the cross bar. The beam was suspended by 2 glass fibres drawn out to 6 μ diameter at their bottom ends (which were joined to the cross bar) from an adjustable support fitted to the top of the balance case. A fused quartz torsion fibre, cemented to the mid-point of the beam, 12 μ in diameter, was joined to a steel torsion drive outside the case and provided the means of measuring the change in weight of the specimen. The counter poise weight at one end of the beam was free to fall a limited distance. A projection microscope system was used to indicate the position of the beam. The operating conditions of the balance are given in the table reproduced below. The case housing the instrument was inserted in a thermostatically controlled oven and could be evacuated by means of a mercury diffusion pump to a pressure below 10^{-4} mm. Hg. The balance was calibrated at the fixed load of 0.1 g. Details of the calibration method are given. The balance indicated reliably a change of weight of 0.02 μg.

Operating Conditions of the Balance

Period of Swing	10 sec
Range of Torsion Fibre	300 μg
Sensitivity of Torsion Fibre	8°/μg
Sensitivity of Balance	2×10^5 radians/g
Screen Sensitivity	50 mm/μg

Sommaire : Un modèle de micro-balance sous vide constituant une amélioration de la balance conventionnelle à ressorts à boudin en quartz fondu est décrit.

Article by A. G. Day *J. Sci. Instrum.* **30**, August 1953 260-263

A Needle Valve-Type of Variable Gas Leak for Mass Spectrometers

See Abstract No. : 164/II

154/II

A Vibrating Variable Gas Leak

See Abstract No. : 194/I

155/II

A Double-Replica Method Using Carbon Layers

156/II

Germany. The metal surface to be investigated is covered with a layer of evaporated silver at least 5 μ in thickness. This layer is easily removed from the surface and is then covered with a thin layer of silica, or better, carbon. The silver is dissolved in 40% nitric acid and the positive carbon replica transferred to the specimen carrier of the electron microscope. (*Science Abstracts*)

Sommaire : Une technique d'une réplique d'un spécimen comportant l'emploi de film de carbone est décrite.

Article by V. C. Dalitz & J. A. Schuchmann *Optik* **10**, Nos. 1-3, 1953 143-146

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II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

A Replica Technique in the Study of Chemical Precipitation Processes — 157/II

United States. Where precipitates formed by mixing solutions of chemical reagents are to be studied in the electron microscope the use of replica techniques is preferable to a direct examination of the specimen. If exposed to the electron beam the genuine specimen material tends to decompose or to be contaminated by residual gases in the chamber. Fischbein developed a 3–step wet replication process for use with biological specimens. The process described in the present article is a 1–step replication process based on Fischbein's technique. A sandwich is prepared consisting of 2 glass slides holding a drop of the mother liquid with the precipitate in suspension between them. One of the glass slides is covered with a film of parlodion. A parlodion film of suitable thickness is obtained by dipping the glass slide into a 3% parlodion solution in amyl acetate and subsequently drying it. The 2 glass slides are clamped together and the sandwich is heated in the mother liquid at boiling point temperature for 10 minutes. The sandwich is then taken apart, the precipitate dissolved, the replica floated off the glass slide on water and picked up by screens in the conventional way. In order to obtain good contrast shadowing is essential. The most suitable angle for shadowing was found to be 30°. Micrographs of directly viewed specimens and replicas of the same material, *e.g.* silver chloride particles, are shown in illustrations.

Sommaire : Une nouvelle méthode de réplique basé sur la méthode de réplique humide de Fischbein des spécimens biologiques est décrite.

Article by R. B. Fischer & J. P. Ellinger *J. Appl. Phys.* **24**, Aug. 1953 1051-1053

Microstructure of Capacitor Paper — 158/II

See Abstract No. : 209/I

An Electron-Micrograph Study of Oxide Films on Electropolished Surfaces of Iron — 159/II

See Abstract No. : 250/I

26 — MATERIALS — 26

The Use of Silicone Oil as an Operating Fuel for Diffusion Pumps — 160/II

See Abstract No. : 131/II

Electrically Heated Glass for Airplane Cabins — 161/II

United States. The type of electrically conducting glass considered in this note consists of 2 glass layers separated by a vinyl plastic which carries a film of stannic oxide, some 20 millionths of 1 inch thick. In order to avoid cracking of these glass panels when exposed to normal weather conditions, the conductivity of the stannic oxide film must be even throughout the panel. To test the performance of the film the panels are placed in a ' Strato-Chamber ' normally used for high altitude research, and operated in flight conditions encountered in practice. The average power consumed, the power at a control point and the power at the hottest point of the panel are then determined and as a result the heating pattern of the panel is established mathematically. The ratio of power at the hottest point to that at the control point should not exceed 1.3 and the temperature of the front surface facing the wind should not drop below 40°F, while the hottest point inside the windshield should not exceed 160°F.

Sommaire : Des problems associés avec l'emploi du verre chauffé électriquement dans la construction aeronautique sont discutés.

Note by R. Bloomberg *Ceramic Age* **59**, May 1955 41

A Vacuum Microbalance for Measuring Sorption in Dielectrics — 162/II

See Abstract No. : 153/II

28 — APPARATUS COMPONENTS — 28

Isolation Valves for Vacuum Systems — 163/II

United Kingdom. The chief types of vacuum valve used for isolating one part of a vacuum system from another are classified and many of the designs published in the last twenty years are reviewed. The requirements of a vacuum valve are considered and criticisms and comments made regarding existing types of valve. Vacuum valves developed from existing valves as used in routine engineering practice as well as valves designed specifically for low pressure work are described. An account follows of recent work on valves for ultra high

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vacuum systems. Possibilities for future designs, chiefly of the type of valve which can be degassed by baking, are briefly discussed.

(*Author*)

Sommaire : Examen des plus importants types de valves méchaniques pouvant être employés dans les appareils à vide.

Article by J. Yarwood *Vacuum* **3**, Oct. 1953 398-411

A Needle Valve Type of Variable Gas Leak for Mass Spectrometers

164/II

United States. Gas leaks of the type employed in mass spectrometers for control of the relation between the gas pressure prevailing in the ion source and that of the sample to be analysed can be classified as fixed or variable or, in terms of flow, as molecular or viscous flow leaks. The variable type of leak covers normally a wide range of sample pressures and provides for viscous flow through the leak. The authors report on the design of a leak which can provide for viscous or molecular flow, and which covers a very wide range of flow rates with the added possibility of being shut-off altogether and thus simplifying the design of gas inlet systems for mass spectrometers generally. The leak is fabricated from a standard Hook 411-type of needle valve and employs the original valve body, diaphragm and needle. The standard spring arrangement is strengthened by an additional helical spring exerting a force of about 45 lbs and a stem positioning cap is substituted for the spring-retaining ring. A steel bar is fitted to the top of the frame and a second bar is hinged to the side of the frame. The sides of the bars facing each other are ground and the bottom face of the lower bar rests against the stem positioning cap. Both bars are in a position parallel to each other and are separated by a steel block which has a thickness 0.015 inch larger than the distance between the two bars. This steel block can be moved by means of a propellor shaft which is passed through the side of the frame. When the block is moved inwards the lower bar will close the valve. In intermediate positions of the bar the size of the leak is determined by the location of the stem positioning cap. Various alternative driving mechanisms are discussed. Leaks of this design have been in use for up to 3,000 hours. At common mass spectrometer pressures, flow through a leak of this design is molecular. By inserting a length of capillary tubing on the high pressure side of the valve the flow characteristics could be changed to those of a non-fractionating viscous flow leak.

Sommaire : Détails donnés sur un nouveau système de fuite variable employé sur un spectrographe de masse.

Article by E. F. Babelay & L. A. Smith *Rev. Sci. Instrum.* **24**, July 1953 508-510

A Leak Control Tube

165/II

United States. In high vacuum work it is often desirable to provide for a pumping impedance of known conductance value which could be varied to an impedance of other known conductance value, without breaking the vacuum. A leak control tube which meets these requirements and has been specially designed for experimental determinations of gettering rates is described in this note. It essentially consists of a Kovar collar $2\frac{1}{8}$ inch in diameter, sealed to a 7052 glass tube. The Kovar collar is closed by a stainless steel plate, brazed onto it, which carries two uprights supporting a threaded molybdenum rod. One end of the molybdenum rod is fitted with a steel block (within the envelope) and can be caused to rotate by means of a magnet. A small stainless steel disc fitted to the rod then moves across a series of holes in the stainless steel plate brazed to the collar. In the particular case, four holes are provided in the stainless steel lid with a diameter of 0.079, 0.129, 0.248 and 0.440 cm. respectively, the corresponding conductances of which have been calculated for carbon dioxide at room temperature to be 10.4, 41.3, 234 and 990 cm^3/sec. The whole assembly is fully enclosed in a glass envelope, and is inserted in the system between the tube containing the getter and the manifold of the pumping system. When all four holes are covered up by the small stainless steel disc the conductance between the disc and the plate is approximately $2\frac{1}{2}$ cm^3/sec. at a pressure of about 10^{-5} mm. Hg, but this comparatively large leakage has little effect on results where two or more holes are used in the experiments.

Sommaire : Déscription d'un tube des fuites controllées, spécialement developpé pour l'emploi dans les expériences sur les getters.

Note by J. Morrison *Rev. Sci. Instrum.* **24**, July 1953 546-547

A Vibrating Needle Variable Gas Leak

166/II

See Abstract No. : 194/I

A Stainless Steel Trap for a Metal Vacuum System

167/II

Belgium. Using a method which has been effective in glass traps a spiral of stainless steel ' corrugated ' plate was introduced into a metal trap. This reduces the consumption of liquid air.

Sommaire : Piège en acier inoxydable spécialement developpé pour limiter la consommation de l'air liquide.

Letter by E. Thomas, J. Destappes & J. Dupont *Vacuum* **3**, Oct. 1953 413-414

A Quick-Change Seal Opener

168/II

United States. In the case of the ORNL 86-inch. cyclotron the target assembly is water-cooled and designed to be rotated or displaced in a linear direction. The stem of the assembly terminates in a water header which is fitted with three Wilson-type seals made from Koroseal gaskets. Exchanging target assemblies is rather time-consuming as one of the seals in the header is in a position opposed to the direction of inserting the stem. The seal opener described in this note is designed to simplify insertion of the stem. It consists of a nut inserted into the seal body in a position concentric with the cylindrical bore accommodating the target stem. The nut terminates in a thin walled sleeve. Rotating the nut half a turn, the upper end of the sleeve travels about $\frac{1}{8}$

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inch inwards and bends the inner edges of the seal gaskets upwards so that the stem can be inserted without shearing the edges of the gaskets. After insertion of the stem the nut is retracted and the gaskets are in their normal operative position. The new arrangement reduced the time required for changing the target assemblies to 5 minutes equal to 1/5th of the total time required previously. The effectiveness of the seals in question is unchanged.

Sommaire : Un obturateur à changement rapide a été développé pour permettre de réduire les pertes de temps d'opérations sur cyclotron.

Note by W. R. Smith *Rev. Sci. Instrum.* **24**, Sept. 1953 886-887

Improvements r.t. the Metallic Lubrication of Ball Bearings

169/II

United Kingdom. Ball bearings are frequently required for use in evacuated apparatus for instance in the construction of rotating-anode X-ray tubes. Normally lubrication of such ball bearings is effected by coating the balls with a layer of a metal or metal compound which varies in thickness from 1 to 8 μ. Application of these coatings by chemical or mechanical means has not proved satisfactory. There is a tendency of flakes forming in operation which break off, resulting in irregularities of surface thickness and texture with attendant deterioration of the lubricating effect. It is claimed that the lubricating performance of coatings can be improved by a method described in this patent. It is recommended to use a compound as the coating material containing lead or tellurium up to a total of 0.1%, or a silver-thallium alloy containing about 2% thallium, or a brass type of metal compound. Two methods of electro-depositing the coating on the balls are described. In one case a rimmed dish, conical in section, containing the balls is fitted to a spindle driven by a small motor and immersed in the bath at an angle in the vicinity of a fixed anode located in the bath, the dish forming the negative electrode. Due to the rotation of the dish the balls constantly move and receive a uniform coating over the whole of their surface area. After completion of the coating the balls are assembled in the races and are heat-treated in a hydrogen atmosphere at reduced pressure. The length of treatment depends on the material of the coating. The heat treatment has the effect that the occluded gases are removed and that intimate diffusion of the various metal components takes place within the coating. This heat treatment may be repeated in order to keep the lubricating performance of the coating at the required standard.

Sommaire : Un procédé pour l'apport d'un film métallique lubrifiant sur les roulements à billes supportant un ensemble mécanique employé dans un système sous vide est décrit.

Newton Victor, Ltd. *Brit. Pat.* 676,902

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29 — MISCELLANEOUS APPARATUS — 29

A Vacuum Anemometer

170/II

United States. There are no devices readily available for the measurement of the projective pressure of vapours arising from the surface of a liquid, boiling at reduced pressure. The authors describe the design and performance characteristics of a vacuum anemometer for the pressure range of 0.1 to 100 micron Hg which was to assist in the study of the surface habits of liquids. Surveying existing means of pressure-sensing elements it was decided to employ the propeller principle as this would provide the simplest solution. In its final form the instrument consists of a propeller mounted on a pivot 0.75 inch long and 0.035 inch diameter made from K–Monel metal with osmium–alloy tips. The tips were held in synthetic sapphire vee-jewels. Two discs, 0.25 inch diameter and 0.062 inch thick, of magnetic material were fitted to the pivot. Two stationary silver discs, 0.44 inch diameter and 0.03 inch thick, were assembled in a concentric position. One disc was placed between the two magnets and 1 disc underneath the lower magnet. Finally, a stationary coil wound on a Bakelite reel and consisting of about 175 feet of copper wire about 0.001 inch diameter was situated near the upper magnet and connected to a voltmeter. The propeller was made from Chromel–A sheet 0.004 inch thick and 1 inch diameter. It had 3 blades each 0.25 inch wide and twisted about 45° near the hub. At constant speed the operation of the arrangement is described by

$$T_d = T_b + T_f$$

where T_d is the driving torque and can be expressed by

$$T_d = p \frac{2 \pi r_0^3}{3} \sin \theta = C_1 p$$

T_b is the braking torque due to the metal discs and can be expressed by

$$T_b = C_2 B' \omega / R$$

By combining these formulae the operating equation becomes

$$E = \frac{C_4 R}{B} (C_1 p - T_f)$$

where E is the emf induced and recorded on the voltmeter, C_4 and C_1 constants dependent on the design of the apparatus, R the resistance of the electrical circuit, T_f the total frictional torque, B the density of the magnetic flux and p the vapour pressure to be determined. As long as the frictional torque is relatively small the indication of the instrument is directly proportional to the pressure and independent of the nature of the vapours and their molecular weight. The instrument is actuated by the difference in transfer of momentum on opposite sides of the propeller. A calibration method is described which is particularly convenient in the case the instrument is to be used in experiments with condensable vapours. In the particular case the test fluid was di–2–ethyl hexyl phthalate. It was known from previous experiments that pure vapours of this liquid are evolved at equilibrium pressure if the surface is continually renewed and of an area some 500 times

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Contd.

Abstract No. and References

that of the orifice from which the vapour escapes. Accordingly the vapour generator built for the purpose consisted of an inverted 14–inch. bell jar placed into an air bath which ensured that the walls were kept at the temperature of the vapour. The liquid held inside the bell jar was sprayed onto the walls of the jar by 2 rotating aluminium tubes. The vapour escape had a diameter of 1.4 inch, and the ratio of area of evaporation to area of the vapour escape was about 600 : 1. The instrument was placed within the vapour escape. After passing the instrument the vapours were condensed in a condenser. During the calibration run the revolutions of the propeller were measured stroboscopically at speeds above 120 r.p.m. and visually below that value. The speeds determined were converted to emf assuming that the voltmeter had a linear response. It is essential that during operation the instrument is held at a temperature slightly exceeding that of the vapours. This is achieved by infra-red radiation. In a series of experiments with this arrangement determinations have been made of the friction torque examining the effect of the various components of the assembly on the total friction value. Detailed results are given for the driction obtained when no braking discs were employed and for the case that only a single braking disc was employed.

Sommaire : En rapport avec les expériences sur le comportement des surfaces liquides s'évaporant sous basses pressions un anemomètre a été développé et des détails en sont donnés.

Article by D. J. Trevoy & W. A. Torpey *Rev. Sci. Instrum.* **24**, Aug. 1953 676-682

Vapour Pressures of Pyridine and Its Homologues 171/II

See Abstract No. : 237/I

A New Type of Helium Liquefier 172/II

United Kingdom. Some considerable time ago it was proposed to liquefy helium or hydrogen by a method similar to that used by Claude for the liquefaction of air, *i.e.* an apparatus operating on the ' external work ' principle and replacing the cylinder and piston of the expansion engine by a bellows arrangement. Experiments with this type of liquefier have been resumed recently after it had been established that the fatigue life of the bellows increases as the temperature decreases and is long enough to be of use in practical work. The bellows employed in the experimental liquefier had the following data: 7.7 cm. outer diameter, 4 cm. inner diameter, 5.4 cm. length, convolutions : 15, material : Brass. One end of the bellows was closed and the other fitted with a cup housing the valves. The cup is fixed to a stainless steel tube in the centre of the vacuum container. As the gas admitted to the bellows expands the lower end of the bellows moves downwards. This facilitates the operation of all controls by means of a rod mechanism actuated by the movement of the bellows. The stroke of the bellows is 2.7 cm. The engine operates at 2–3 cycles/second and at the operational pressure of 5.5 atmospheres, the life of the bellows is approximately 500,000 cycles. The vacuum case which also contains the heat exchangers and the liquid helium container is immersed in a glass Dewar vessel which contains liquid air. The apparatus produces 0.4 l. of liquid helium per hour and consumes 1.2 l./hr. of liquid air.

Sommaire : Un appareil à liquefier l'helium dans lequel le cylindre et le piston habituellement employés dan le système d'expansion sont remplacés par un soufflet métal élastique a été décrit.

Letter by H. M. Long & F. E. Simon *Nature* **172**, 26.9.1953 581

Apparatus for Evacuating Hollow Articles 173/II

United States. Mass-produced articles such as electric tungsten and discharge lamps or thermionic valves have to be evacuated at one stage of their production. Normally a batch of these components are exhausted simultaneously. If any one component in the batch has, or develops, a leak it has to be eliminated from the production batch in order to avoid contamination of the other components in the batch. The conventionally used mechanism for the detection of a leaky component in the batch is a mechanical pinching device which operates during the rough-pumping stage and is actuated electrically by a relay which is controlled by the pressure rise in the leaky component. This device has the disadvantage that it only detects comparatively large leaks, noticeable in the roughing stage. The device proposed in this patent, however, operates in the fine vacuum pumping stage and facilitates the detection of small leaks which may do just as much harm. The new device consists of a valve which is inserted between every pumping head and the vacuum pump of the exhaust machine employed for this type of process. The valve chamber has two apertures, one to be connected to the respective pumping head of the machine and one to the tubulation leading to the pump. It also contains a free-moving metal ball. This ball, in its normal operational position, rests on a shelf in front of, and inclined to the aperture connected with the pumping head. In front of the second aperture leading to the vacuum pump is a plug bored out to register with this aperture and fitted with an ' O '–ring on the inside of the chamber. If the ball comes to rest on this ' O '–ring the pumping line is blocked. As the component to be pumped is moved from the point of assembly on the machine to the rough pumping stage the ball resting in front of the first aperture is lifted by external magnetic action, thus facilitating unobstructed passage of the gases pumped through the valve chamber. When the component has progressed to the fine pumping stage the ball is released by the magnet and drops into the resting position. In the normal course of pumping so small an amount of gas is passing through the chamber at this stage that the ball will not be moved from this position. In the event, however, the component pumped has a leak, the quantity of gases passing through the chamber will increase rapidly and will cause the ball to move to the position in front of the outlet to the pump, thereby sealing off this component from all communications with the rest of the system. The defective component can then be removed without affecting the pumping action in any of the other components processed simultaneously. It is an essential part of the invention that the valve body is made from non-magnetic material.

Sommaire : Un dispositif facilitant la détection des fuites et l'isolement du tube ayant une fuite, fixé sur les canalisations d'une station de pompage, faisant le vide dans une série de tubes thermo-ioniques est décrit.

Sylvania Products Inc. *Brit. Pat.* 697,069

III — Vacuum Processing Techniques — III

Abstract No. and References

30 — EVAPORATION — 30

High Vacuum Evaporation 136/III

United States. Evaporating 2 mutually miscible non-metallic materials from a common crucible at reduced pressure, vapours are generated which contain the two materials in a ratio which is related to the ratio of the solids forming the original charge by the phase diagram for the operational pressure. An analysis of the vapour can easily be effected as the deposit obtained is of the same ratio as the vapour phase. Thus in a series of experiments it is possible to collect data for the liquid-vapour phase diagram. The information is often needed in the deposition of anti-reflection coatings where the latter are required to have a refractive index inbetween the values of the refractive indices of the 2 pure components. The technique could also be applied in connection with evaporation of 2 metallic materials, for instance for the production of metal-alloy mirrors.

Sommaire: Avis est donné sur la façon de procéder la déposition des couches anti-réfléchissants sous vide lorsque un indice de réfraction intermédiaire entre ceux de deux composants pures est demandé pour le depôt.

Letter by J. E. Tyler *J. Opt. Soc. Amer.* **43**, August 1953 708

Metallising of Glass, Ceramic and Plastic Surfaces 137/III

United Kingdom. Three basic methods of depositing metal films on the surfaces of materials are discussed, *i.e.* reduction from aqueous solutions, reduction by heat, and vacuum evaporation. The deposition of silver films by the first method can be effected by cane sugar reduction, formaldehyde-Rochelle salt reduction or by hydrazine sulphate reduction. Details of the composition employed are given. The Rochelle salt reduction gives the best results so far as adhesion is concerned. The production of copper, gold and antimony deposits by this method is described. The method of reducing metal compounds by heat is limited to materials which have a sufficiently high heat resistance on one hand, and to metals, the compounds of which are reducible by heating on the other. Suitable materials are the salts of silver, gold, platinum and palladium and suitable substrates are ceramics. Details of the deposition of silver, gold and platinum by this method are given. Finally the basic facts are given on the method of depositing metal films by vacuum evaporation. The thin films produced by any of the 3 methods can be thickened by means of electrodeposition. In order to prevent chemical attack the thin films are first treated in a special plating bath before they are transferred to the normal bath. In the case of silver films it is recommended that the special bath consists of silver nitrate (25 g.), sodium cyanide (28 g.), potassium nitrate (100 g.), and water (1 litre.). The current density should be 0.5 A/sq. dm. Silver anodes should be used and the temperature of the bath should not be below 25°C. After the deposition of 0.0005 inch of silver the part can be transferred to a bath of the normal composition. Similar recommendations are made for the coating of copper and nickel by electrodeposition. A typical application of the method of reduction from aqueous solutions is the deposition of silver on insulating materials to provide a conducting layer for uses in electro-forming practice. The firing method is used for decorative purposes on pottery and lately also in the manufacture of resistances. Applications for the evaporation method shown are: Mirrors, selenium rectifiers and photocells.

Sommaire : Un examen général concernant toutes les méthodes connues de métallisation sur verre, céramique, et surfaces de matière plastique.

Article by R. J. Heritage & J. R. Balmer *Metallurgia* **47**, April 1953 171-174

The Effect of the Substrate Temperature on the Condensation Coefficient of Evaporated Antimony, Gold and Silver 138/III

See Abstract No. : 221/I

On the Structure of Thin Silver Layers 139/III

See Abstract No. : 220/I

Capacitance Effects in Thin Conductive Films 140/III

United States. The frequency dependence of the electrical resistance of thin films of lead sulphide and tellurium was compared with the frequency characteristics of models consisting of suitable resistance capacitance networks. The model corresponding to microcrystalline structure was represented by a network of intercrystallite resistance and capacitance and the internal resistances of crystallites while the model of a homogeneous resistor was represented by a network of distributed resistances and capacitances. Formulae derived by Chasmar of the equivalent parallel resistance capacitance network for the microcrystalline model and by Howe for the distributed model were used in this paper. The models were also interpreted for temperature dependence. In the case of a model of predominantly intercrystallite capacitance the temperature effect should be more marked. Measurements of the resistance *versus* frequency characteristics and its temperature dependence for 2 tellurium films and 1 lead sulphide film were compared with the frequency characteristics of equivalent models. A tellurium film fitted the concept of distributive capacitance while the other films could best be fitted to a model based on a combination of homogeneous and microcrystalline structure. Both types of film showed photoconductive properties.

Sommaire : La relation entre la structure du film et les effets capacitifs de couches de sulphure de plomb et de tellurium est étudiée.

Article by J. N. Humphrey, F. L. Lummis & W. W. Scanlon *Phys. Rev.* **90**, April 1953 111-114

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

Measurements of Electrical Resistance on Thin Iron Films

141/III

Belgium. Recent research work on the electrical resistance of thin iron films is reviewed with particular reference to the studies of Mostovetch. It appears that the original conception explaining the resistance properties of thin metal films in terms of mean-free-path conditions is unsatisfactory. This is attributed to the fact, now established with the help of electron microscopy, that the films have a granular structure. However, a reproducible quantitative assessment is still difficult as the granular structure is influenced by the method employed to form the film. Fluctuations in temperature during and after formation and the presence of impurities in the deposit can modify the structure and thus the resistance characteristics. For this reason films to be used in this type of experimental work should preferably be deposited by vacuum evaporation and not by cathodic sputtering as the latter process is more difficult to control. Large negative temperature coefficients occur when the average thickness of the film falls below a certain limit. Also, films in that range of thickness show deviations from Ohm's law which are the greater, the lower the temperature. Another factor, not yet under control and affecting the accuracy of experimental results, is the variation of distance between, and size of, the individual granules in the films. Mostovetch gives a formula for the conductivity of the film in terms dependent on the distance between the granules and for the reason stated is not applicable generally. Gorter gives a formula based on the conception of the granules forming small condensers but this approach, too, appears to be of limited application as it seems to be correct only, where the distance between the granules is relatively small. The author concludes that a proper explanation of the resistance mechanism of thin metal films cannot be expected, until the film structure has been more fully explored.

Monograph by S. R. Lambeir *Ver. Koninkl. Vlaamse Acad. Wetensch.* No. 40, 1953 97 pp.

Sommaire : Les résultats des travaux de recherches sur la résistance éléctrique de couches minces de fer sont donnés.

Electrically Heated Glass for Airplane Cabins

142/III

See Abstract No. : 161/II

Electron Currents in Thin Oxide Films on Aluminium

143/III

See Abstract No. : 248/I

Thin Metallic Films for Anti-Reflection Coatings on Dielectric-Air Interfaces

144/III

United Kingdom. Experimenting with vacuum-deposited rhodium films on glass, it was noticed that the reflection intensity obtained at the coated glass-air interface was appreciably smaller than that obtained after reversing the glass plate. Investigating this matter further a wedge-shaped rhodium film was deposited on glass of 1.52 refractive index and it was found that at a thickness of the film giving 50% transmission the reflection was less than from the bare glass surface, and at 70% transmission it was virtually zero. This effect can be used with advantage in light microscopy where examination of the rhodium surface by normal bright-field vertical illumination gives a dark-field image showing up brightly chemical film patches, etc. on the surface.

Letter by F. H. Smith *Nature* **172**, 5.9.1953 462

Sommaire : Des informations sont données sur les propriétés anti-réflection d'un verre metallisé avec un film de rhodium.

Improvements i.o.r.t. the Reduction of Reflection from the Surface of an Article

145/III

Lichtenstein. The reduction of reflection at a glass surface is effected by the provision of a thin film causing interference action. This effect can be increased to any required degree by providing a multiple film where at least one of the layers must consist of a material of high refractive index. Such multiple anti-reflection films can be produced by cathodic sputtering or by decomposition in the vapour state, but both these methods involve the use of high temperatures. Deposition of such films by the evaporation method would solve the temperature problem but in practice it has failed, so far, to give good results because highly light-absorbing mixed oxides are formed in the deposit. A method is described whereby the evaporation process can be used with success for the deposition of such films. It is based on the use of materials which belong to the rare earth group of elements, with atomic numbers 58-71 inclusive. It has been found that cerium oxide (CeO_2) for instance, gives an absorption-free deposit with a very high refractive index and it is proposed, therefore, to build up multiple films using several metal fluoride interference layers which are known to be of low refractive index, and at least one cerium oxide film deposited by vacuum evaporation in the same manner as the low refractive index-layers.

A. Vogt *Brit. Pat.* 697,403

Sommaire : Une méthode de préparation de couches anti-réflection durables par moyen d'évaporation sous vide est donnée.

Review of Work on Dichroic Mirrors and Their Light-Dividing Characteristics

146/III

See Abstract No. : 212/I

Thin Layers Transmitting Short Ultra-Violet Rays

147/III

See Abstract No. : 226/I

III — Vacuum Processing Techniques — III
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Abstract No. and References

Narrow Pass Band Optical Filters 148/III

See Abstract No. : 223/I

An Inspection Method for Luminescent Screens 149/III

See Abstract No. : 211/I

Metallic Grating Replicas as Internal Standards for Calibrating Electron Microscopes 150/III

United States. It was shown by J. H. L. Watson and W. L. Grube previously that grating replicas made from plastics and supported by $\frac{1}{8}$ inch specimen screens can be used as internal calibration standards for electron microscopes. Plastics replicas have a tendency to shrink. Therefore all-metal replicas would be preferable but normally their production involves the destruction of the specimen. The authors report on the manufacture and performance of all-metal grating replicas without affecting the original grating surface. The method employed is similar to that described by W. L. Grube and S. R. Rouze (*Proc. Am. Soc. Testing Materials*, **52**, 1952, 71). A layer of Victawet 35 B is deposited on the original grating by vacuum evaporation and serves as the parting layer in the stripping operation. The grating is then shadowed with chromium at 27° in a direction perpendicular to that of the rulings. On completion of pre-shadowing, a layer of 45 : 55 aluminium-beryllium alloy is deposited by evaporation in the normal way. The replica is then direct-stripped from the specimen by floating it off on a water surface and picking it up on a 720-mesh copper screen. The 720-mesh screen was required as an all-metal replica is more fragile than a plastics replica. The spectrogram of an all-metal replica of a 15,000 lines/inch. grating, obtained during calibration of the replica as a transmission grating in a Gaertner spectrometer using a mercury arc lamp, is shown in an illustration. In these calibrations the average d value of the replicated grating varied from the original grating by less than 0.3%. Measuring the grating replica in the electron microscope at a magnification of 5,360x the deviations of the readings were about the same as in the case of plastics replicas.

Sommaire : Description d'une technique de réplique métallique des écrans de diffraction.

Article by S. R. Rouze & J. H. L. Watson *J. Appl. Phys.* **24**, Sept. 1953 1106-1109

A Double-Replica Method Using Carbon Layers 151/III

See Abstract No. : 156/II

Microstructure of Capacitor Paper 152/III

See Abstract No. : 209/I

31 — CATHODIC SPUTTERING — 31

Cathodic Sputtering an Analysis of the Physical Processes 153/III

Germany. The author discusses the results obtained from studying the sputtering effect, first discovered nearly one hundred years ago. He introduces the concept of primary sputtering, subdivided into first and second mechanism, and secondary sputtering. Various factors determining the rate of sputtering, using different metal-gas combinations, are investigated. Secondary sputtering, normally observed in a glow discharge and the form of sputtering essentially of technical interest, is defined and the limits of measurement are described. In conclusion, the author remarks on 'chemical' sputtering, on certain engineering aspects and, finally, on gas adsorption, a phenomenon inherent in the sputtering process.

Sommaire : Article détaillé sur les connaissances physiques de projections cathodiques.

Article by A. Guenther-schulze *Vacuum* **3**, Oct. 1953 360-374

The Properties of Some Reactively Sputtered Metal Oxide Films 154/III

United Kingdom. Some of the recent applications of reactively sputtered metal oxide films for electrical and optical purposes are described. A series of conducting oxides approximating to the compositions CdO, SnO_2, In_2O_3 and FeO have been prepared and their respective properties are discussed in relation to their use as transparent conducting coatings on glass windows. It is shown that the electrical conductivity of sputtered CdO–films decreases as the degree of oxidation of the deposit rises. The conductivity of a highly oxidised deposit can be greatly increased by heating the film in air to cause a partial dissociation of the oxide. Cadmium oxide films of the highest conductivity may also be produced directly by careful control of the glow discharge and residual gas mixture during sputtering. The method of preparing gold films of compact structure by using bismuth oxide substrates due to Preston and Gillham has been investigated, and their results are confirmed. Gold films of 100 Å thickness on bismuth oxide are shown to have a low resistivity (10 ohms per square) and a high optical transmission (75 per cent.). Values of the resistivity and temperature coefficient of gold-films on bismuth oxide are given for films 20–400 Å thick in the temperature range 15–150°C. It is shown that the temperature coefficient decreases with the film thickness and can be related to a rise in the resistivity of the deposit. The optical transmission and resistivity of typical coatings of each of the above materials is given in tabular form.

Sommaire : Des appareils pour la production et les applications de films d'oxyde de métal déposés par projection réactive cathodiques sont décrits.

Article by L. Holland and G. Siddall *Vacuum* **3**, Oct. 1953 375-390

III — Vacuum Processing Techniques — III
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Abstract No. and References

Reactive Sputtering and Associated Plant Design 155/III
See Abstract No. : 198/I

Electrically Heated Glass for Airplane Cabins 156/III
See Abstract No. : 161/II

33 — IMPREGNATION — 33

Solvent Recovery from Pentachlorophenol-Treated Wood 157/III

United States. A new method of preserving wood has recently been introduced which provides for the impregnation of the timber with pentachlorophenol. The use of this material without any solvent is not practical as the material would not penetrate the timber to any appreciable depth. The choice of a suitable solvent has proved difficult. Some tend to produce a ' blooming ' effect on the surface of the wood, when the solvent has evaporated, and others cause ' bleeding ' to the surface. It was decided, therefore, to investigate means of modifying the impregnation process whereby the solvent used is completely removed after completion of the treatment. In the normal ' vapour-drying ' process green wood is loaded into the pressure-treating cylinder and exposed to vapours from an organic solvent boiling in an evaporator. The vapours condense on the wood, heat the wood at the same time and cause the moisture to evaporate from the wood. The resultant vapour mixture of water and solvent is conducted to a separator where the water is discharged and the solvent returned to the evaporator. The recovery of the solvent (xylene in this particular case) is effected as follows : After completion of the impregnation process xylene only is boiled in the evaporator at 280°F and the vapours produced are passed over the wood in the cylinder. After about 1 hour the wood has reached the temperature of the vapour. During that period some of the xylene condenses on the wood, drips off the wood and is collected in a condensate tank. At the end of the first hour the evaporator is shut off and a vacuum of about 25 inch. is applied which causes the xylene, which had entered the wood during impregnation, to evaporate. Repeating this cycle of 1 hour heating and 1 hour vacuum results eventually in the complete removal of the xylene from the wood. Apart from xylene, high-flash coal tar naphtha, perchloroethylene and trichloroethylene have been found to be solvents suitable for this form of treatment. The author gives details of experiments carried out with xylene as the solvent under varying conditions. Detailed results are shown in a table. In experiments with 5% pentachlorophenol solution impregnation was carried out initially at a pressure of 125 lb/squ. inch, maintaining this pressure for an hour, following which the cylinder was evacuated and kept at reduced pressure for another hour. After additional treatment for solvent recovery in the manner described the solvent content in the wood was reduced to 0.1 lb./cu. ft. of dry pentachlorophenol. As this figure was below 0.25 lb./cu. ft., which was the residual solvent content aimed at, a second experiment was conducted using a vacuum of 22.5 inch. during the impregnation process. In that case the amount retained was found to be excessive. Satisfactory results were finally obtained using a pressure of 25 lb./squ. inch during impregnation after increasing the pentachlorophenol concentration to 10%.

Sommaire : Un bois impregné de pentachlorophenol montre des taches dues aux solvants volatiles employés. Un méthode évitant cette difficulté est décrite.

Article by M. S. Hudson *Industr. Engng. Chem.* 45, July 1953 1576-1583

34 — FUMIGATION — 34

Descriptive Terms for Vacuum Fumigation 158/III
See Abstract No. : 188/I

36 — DRYING — 36

Evaporation 159/III

United Kingdom. The author surveys recent development in the field of evaporation processes and evaporator design. Reference is made to recent descriptions of the production of ammonium nitrate by three different methods. A table reproduced below gives the comparison of these methods. The prilling process consists essentially in spraying hot concentrated ammonium nitrate liquor from the top of a tower through which a stream of air at a lower temperature is rising. Evaporation is carried out in a stainless steel evaporator of the long-tube single-effect recirculation type removing approximately 2,000 lb./hr. of water from a charge of 140 ton at a vacuum of 17 inch. Hg, achieving a concentration of 95%. Similar details are given of the vacuum crystallisation process (see also Abstract 214/III, Vol. II). Evaporators, operating at 26 inch. Hg, are used in the production of chemicals from milk. Milk heated at 90°F is passed through solid bowl separators facilitating

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Abstract No. and Referenc es

Table I. Comparison of Ammonium Nitrate Processes *

	Graining	*Crystallising*	*Prilling*
Equipment	Open-pan evaporators, graining kettles, coolers	Vacuum evaporators, crystallisers, driers, centrifuges.	Vacuum evaporator, prilling tower, cooler.
Method	Open-pan evaporation. Graining in stirred kettles.	Continuous vacuum evaporation and crystallisation.	Solidification of strong solution in current of cool air.
Temperature	305 to 310°	160°	260 to 285°
Particle Size	−12 + 35 mesh	−16 + 35 mesh	−8 + 20 mesh
Particle Shape	Rounded granules	Rounded crystals, or cylindrical with rounded ends.	Spherical balls

* Shearon, W. H., and Dunwoody, W., *Industr. Engng. Chem.*, **45**, 1953, 496.

the removal of 10% as high-fat cream. The skimmed milk is treated with hydrochloric acid to give curd. The whey obtained contains 6% lactose, proteins and inorganic salts and is processed with milk of lime to give calcium albuminate. This material is filtered and concentrated in an evaporator with a vaporising capacity of 25,000 lb./hr. to a solution containing 40% solids. Further treatment and additional evaporation yields pure lactose. Similar descriptions are given of the uses of evaporators in the production of lactic acid from corn sugar and in glue manufacture. Reference is made to recent findings by Kelly, (*Chem. Eng. Prog.* **48**, 1952, 589), on the uses of low temperature operation in the field of chemical engineering generally and to a paper by Coulson and Mehta (*Trans. Inst. Chem. Engr.*, 10.3.53) on theoretical and experimental investigations into heat transfer coefficients in the operation of climbing-film evaporators. A new evaporator design is described which consists of a circular tube operated in a vertical position which for 2/3 of its length is surrounded by a heating jacket. The top part of the tube is not jacketed and serves as the separator. A rotor is situated inside the tube which is driven by a motor situated outside and the blades of which move very closely to the walls of the tube. The material to be concentrated is fed into the tube just above the jacketed portion and is thrown onto the walls in the form of a thin film by the action of the rotor blades, where it flows down by gravity becoming concentrated as it falls. The concentrated material is collected at the bottom and the vapours are passed out through the top of the evaporator.

Sommaire : Un article détaillé sur les applications des procédés d'évaporation dans l'industrie chimique.

Article by J. M. Coulson *Chem. Process Engng.* **34**, July 1953 197-200, 222

A Vacuum Drying Apparatus for Small Samples 160/III

United States. The apparatus described is to serve as a substitute for the Fisher pistol, frequently used for the vacuum drying of small samples. The apparatus is shown in an illustration and can be fabricated in a laboratory from the following components : Pyrex tubing 34 mm. diameter, standard taper 35/40 joint, Nichrome wire B and S No. 22, a variable resistance of about 200 ohm., a 3-way stopcock, asbestos sheet, wire gauze and a board as the base. The Pyrex tube is wrapped with 2 layers of asbestos sheet, the sheets serving as envelopes for the Nichrome wire coil. The outer asbestos layer is damp, and the assembly is dried in an oven for 24 hours at 110°. The wire gauze, bent into a suitable shape to serve as the vial carrier, is positioned in the centre of the tube.

Sommaire : Des détails sont donnés sur un petit appareil de laboratoire pour le séchage sous vide.

Note by J. D. Reinheimer *J. Chem. Educ.* **30**, March 1953 139

Continuous Soap Manufacture According to the Mazzoni Method 161/III

See Abstract No. : 186/III

37 — METALLURGICAL PROCESSES — 37

Vacuum Melting Lengthens Fatigue Life, Improves Impact Properties 162/III

See Abstract No. : 256/I

The Technique of Melting and Casting under High Vacuum 163/III

See Abstract No. : 255/I

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Abstract No. and References

Vacuum Melting Developments

164/III

Switzerland. This is a brief survey of recent progress in the design of vacuum furnaces. Four different principles of pouring the metal are shown in a sketch. Two of these make it necessary that the furnace chamber as a whole is tilted, and two provide for bottom pouring : In one case a tapered plug is used and in the second case a plug of the same material as that which is processed. This is melted when required by means of an auxiliary heating coil. A typical time-pressure diagram is shown for the case of melting a charge of 11 lb. of steel containing 0.45% carbon, 1.2% chromium and 0.15% vanadium which was previously melted at atmospheric pressure. The crucible used was of magnesium oxide. Pumping down in the cold state, a pressure of 8×10^{-4} mm. Hg was reached after 4 minutes, then the heating was switched on and melting commenced after another 8 minutes. Due to the evolution of gases, pumping was switched off at this stage for 9 minutes until melting was complete and all additions had been made. Then pumping was resumed for 6 minutes prior to pouring.

Article by Anon. *Metal Industr.* **83**, 25.9.1953 263-264

Sommaire : Note décrivant certains aspects de l'amélioration de modèles de fours à vide.

Induction-Melting Furnaces

165/III

See Abstract No. : 257/I

The Properties of Cast Chromium Alloys at Elevated Temperatures. I. The Melting and Casting of Chromium-Rich Alloys

166/III

United Kingdom. In the course of a general investigation into the properties of chromium-rich alloys special investigations were conducted on methods of producing chromium alloys of low oxygen content. Chromium alloys are produced either by powder-metallurgical methods or by melting and casting. This article is concerned with the latter method. Commercially available pure chromium (electrolytic flake) contains about 0.06% iron, 0.026% sulphur, 0.01% nitrogen and less than 0,01% silicon but its oxygen content is appreciable. Adcock heating chromium in a vacuum at 800°C, demonstrated that oxygen present in the metal combines to Cr_2O_3. The authors repeated the experiments described by Adcock, *i.e.* heated the chromium flake in a vacuum at varying temperatures in the range of 800 to 1,150°C and subsequently dissolved the chromium in hydrochloric acid solution of varying concentration. The insoluble content left in the solution was confirmed to be Cr_2O_3 by X-ray diffraction examination. The actual results obtained in these experiments agreed closely with the figures obtained in parallel determinations using the vacuum fusion method, therefore the authors decided to use the chemical method for oxygen determination in this work. The furnace employed in the experiments is described in full in ' Metallurgical Equilibrium Diagrams ' (Institute of Physics, London, 1952). It consists essentially of a silica tube operated in a vertical position, lined with insulating brick. The crucible is of pure alumina and is surrounded by a sleeve of molybdenum. Above the crucible is a throat made from alumina and held inverted over the throat is the mould. For casting, the furnace is rotated on trunnions. Heating is effected by induction with the molybdenum sleeve acting as the link and evacuation is carried out by a diffusion pump unit. Melting is carried out in a vacuum but casting effected only after argon at atmospheric pressure had been admitted to the furnace. Chromium alloys suffer oxidation even when melted at a pressure as low as 0.005 mm. Hg. A 90 : 10 chromium-cobalt alloy, the material used throughout these experiments, containing initially 2.1% of Cr_2O_3 showed an oxygen content of 2.61% after straightforward vacuum melting. Additional methods, therefore, must be employed to reduce the oxygen content. Treating the melt with purified hydrogen proved to be too slow. A more effective method was used, originally described by Parke and Bens : Carbon is added to the charge which initiates the reaction $Cr_2O_3 + 3C \rightarrow 2Cr + 3CO$. This action is reversible, but at temperatures above 1,250°C it moves towards the right. As the carbon monoxide formed is continuously pumped away the reduction can be allowed to proceed almost to completion. A typical pressure/time curve for the treatment of the alloy mentioned is given. This curve shows various pressure peaks recording the initial evolution of hydrogen when the chromium starts to melt. After about 100 minutes all gas evolution has ceased and reaction is complete. Casting must be timed properly to avoid excess carbon content of the final product, or alternatively, reoxidation of the chromium. Treating the chromium-cobalt alloy by this method the casting shows a residual carbon content of 0.02% and a Cr_2O_3 content of 0.16% = 0.05% oxygen.

Paper by A. H. Sully E. A. Brandes & A. G. Proven *J. Inst. Metals* **81**, 1952-53 569-572

Sommaire : Un procédé pour désoxyder sous vide le chrome et ses alliages est décrit.

The New Metal Titanium

167/III

See Abstract No.: 254/I

Arc Furnace Developed for Casting Titanium

168/III

United States. Normally titanium products are available only in wrought form and are limited to plate, sheet forgings, and some tubing or wire. The machining of titanium and its alloys is difficult and for that reason it is economically justified to make castings of titanium available although this is a complex process. An arc furnace is described suitable for the melting and casting of titanium alloys which has been developed at the Frankford Arsenal. It consists essentially of a gas-tight water-cooled copper crucible and cover, a water-cooled tungsten-tipped electrode with a slight bend at its melting end which is passed through the cover of the furnace in such

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Abstract No. and References

a manner that it can be moved across the bottom of the melting chamber in a circle of about 4 inch. The graphite mould is placed in a cavity in the wall of the melting chamber and is shut-off during melting from the chamber by a retractable door. Melting is effected in the upright position of the furnace, and casting after tilting the entire furnace and retracting the mould door. Castings up to 1½ lb. can be produced. The furnace is evacuated by a diffusion pump unit. Means for admitting argon are provided. The lid of the melting chamber is sealed by O–rings. In operation the melting chamber is evacuated to 1 cm. Hg and subsequently filled with argon. This is repeated several times before the arc is struck which is maintained in an argon atmosphere of a positive pressure of about 3 cm. Hg. In order to ensure uniform composition of the titanium alloy to be cast, pigs of about 2½ lb. each are produced first which contain the desired alloying material in even distribution. Details of the production of the pigs are given. Preparing the final casting from the titanium alloy 5 pigs are placed in the furnace. The furnace is sealed, evacuated and flushed with argon as described and the electrical arc directed to the top of the block in order to melt the upper layer of the charge. Using an arc of about 26 V and 700 A the 5 pigs form a single block, the skull, after about 10 minutes. The current is then increased to about 1,200 A which gives a deeper penetration of heat in the skull, in order to melt enough material to facilitate casting. After about 5 minutes at high current operation the mould door is retracted, the current is turned off and the metal poured by tilting the furnace. The whole operation of pouring takes about 2 seconds. After completion of the pouring the casting is still connected to the parent skull by a metal spine which is severed by re-striking the arc and operating it at 360 A and 23 V moving the electrode into a suitable position. The inclined tip of the electrode is provided especially for the purpose of facilitating this operation. Several castings can be made from the same skull. Pouring can be effected about 20 minutes after striking the arc The moulds which are specially degassed before use, can be employed for 10-15 castings.

Sommaire : Un four à arc sous vide developpé pour la fonte du titane est décrit en détails.

Article by O. W. Simmons & H. R. McCurdy *Amer. Foundryman* **23**, April 1953 121-126

Process for the Purification of Titanium

169/III

United Kingdom. The existing methods of removing impurities from titanium either depend on treatment in the solid state or are based on the reaction of iodine with the metal. In the case of the former method it is difficult to obtain satisfactory results and the alternative method consists of the ' hot-wire ' process which requires costly materials. A new method is described in this patent which is designed to purify substantially pure titanium from impurities combined with oxygen or nitrogen. As a first step the impure titanium is alloyed with another metal, preferably molten zinc. Subsequently, the undissolved material is separated from the liquid alloy and finally the alloyed metal is distilled off leaving the titanium in a pure state. The separation of the undissolved material is effected by filtering the molten alloy through a bed of graded alumina. After filtration the material is allowed to solidify, is crushed to pass a ¼ inch B.S. sieve and then subjected to heating at 850°C (in the case of zinc) for 6 hours at a pressure of 0.001 mm. Hg. It is advantageous to apply the heat slowly so that the material remains solid over the first 2 hours of the treatment. The zinc volatilises during this process and deposits in cooler parts of the plant. 98% of the zinc used is recovered.The final titanium product consists of a non-pyrophoric sintered sponge which after melting has a purity of 99.5%, an oxygen content reduced from 0.8 to 0.2% by weight and a nitrogen content reduced from 0.2 to 0.1% by weight. The zinc content is less than 0.01%. Two alternative forms of procedure based on the same method are described.

Sommaire : Procédé décrit de la purification du titane contenant des combinés d'oxygène ou d'azote.

J. F. Quin & Imperial Chemical Industries Ltd. *Brit. Pat.* 697,487

Apparatus for Attaching Filaments to Electrodes in Machines for Coating with Metal Vapours

170/III

United States. The production of a bar of zirconium, for instance, by the ' hot-wire ' technique, is carried out in a apparatus which is evacuated and fitted with a wire suspended between 2 electrodes, through which a current up to 2,000 A is conducted. The charge in the vessel is heated by external means and iodine vapours are introduced. As a result zirconium iodine vapours are formed which will decompose on contact with the wire as long as the temperature of the latter is kept at about 1,500°C. As a result pure zirconium is deposited on the wire, the iodine is liberated and diffuses back to the charge where it recombines. In the conventional design of this type of apparatus the ends of the hot wire tend to cool down as a result of heat being conducted away as the deposit on the wire grows. As the current is steadily increased during the process in order to maintain the temperature of the wire *plus* the deposit above the lower limit required, the wire has a tendency to fuse at the ends. To counteract this risk it is proposed to change the design of the conventional electrode by adding to it an elongated tip with a central hole, through which the wire is introduced and suitably anchored within. The electrode tip is a detachable component with a large head portion to be connected to the main electrode body and tapered at the other end to a dimension not exceeding twice the diameter of the wire which is normally 0.08 inch. The electrode tip can be made from any refractory material, such as molybdenum, tantalum, titanium or tungsten. In apparatus fitted with the new electrode the junction of the wire and the tapered end of the electrode tip is soon covered with deposit and, as the process is carried on, the deposit continues to form along the tapered electrode tip, thus providing an increased-current carrying deposit, avoiding the risk of overheating the wire and extending the length of the bar formed in the process which is an additional economic advantage. (See also Abstract 171/III.)

Sommaire : Le brevet décrit des moyens améliorés pour le montage des filaments dans le procédé ou les métaux sont precipités par décomposition thermique d'un composé.

Z. M. Shapiro & U.S. Atomic Energy Commission *U.S. Pat.* 2,637,297

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Abstract No. and References

Apparatus for Attaching Filaments to Electrodes in Machines for Coating with Metal Vapours — 171/III

United States. This patent refers to the same technique as that described in Abstract No. 170/III. The particular design of the electrode tip suggested in Abstract No. 170/III may prove to be expensive, in particular as it is normally intended for use on one occasion only. For this reason a more economical design of the electrode tip is proposed. Essential parts of this can be used indefinitely. The modified design of the electrode tip consists of a cup-shaped adaptor which can be slipped over the exposed lower end of the electrode and which is the permanent part. The adaptor has a number of concentrically arranged apertures in which wires are mounted, preferably of unequal length, the outer wires being shorter than the inner wires. The cup and the wires should be of molybdenum material. The ' hot-wire ' is introduced through the central aperture of the adaptor cup and anchored within by suitable means. Two alternative designs of electrode tips based on the same adaptor principle are also shown.

Sommaire : Des détails sont donnés d'une amélioration apportée à l'appareil décrit dans le résumé No. 170/III.

Z. M. Shapiro & U.S. Atomic Energy Commission *U.S. Pat.* 2,637,298

The Micro-Determination of Traces of Gaseous Elements in Metals by the Vacuum Fusion Method — 172/III

United Kingdom. The known methods of gas determination by the vacuum-fusion method normally require samples of a size varying from 5–25 g, from which 2–10 ml of gas are produced and analysed by conventional methods. In an effort to improve and simplify this method, the authors developed an apparatus for vacuum fusion analysis on a much smaller scale. Full details of the apparatus and its operation are given. Five samples could be stored simultaneously and could be analysed successively in one run. The samples were placed into the crucible of the furnace by magnetic means. A graphite crucible was used, shaped in a manner which minimised uncontrolled evaporation from the sample without seriously hindering gas evolution. The crucible was situated in a silica tube and was heated by a high-frequency coil surrounding the tube. Minimum temperature of the furnace was 2,000°C. Gas analysis was carried out by means of the mass spectrometer, or, where the samples exceeded 10 cu. mm. by the Blacet-Leighton method. The new method was applied to the determination of gas content in uranium samples and comparative measurements were taken by means of a macro-method. For the results, see table reproduced below. The uranium samples employed in the micro-

Comparison of the Proposed Method with Sloman's Method for Uranium

Analysis by Micro-Method

Sample No.	*Oxygen % w/w*	*Nitrogen % w/w*	*Hydrogen ml. per 100 g*
1	0.0078	0.0018	0.9
2	0.0095	0.0005	1.1
Mean	0.0087	0.0012	1.0

Analysis by Sloman's Macro-Method

Sample No.	*Oxygen % w/w*	*Nitrogen % w/w*	*Hydrogen ml. per 100 g.*
A	0.0070	0.0010	1.04
B	0.0083	0.0012	0.84
Mean	0.0077	0.0011	0.94

method ranged in size from 120 to 190 mg. An attempt to determine the oxygen and nitrogen content in a zirconium sample was unsuccessful, most likely for the reason that the temperature of the furnace could not be raised high enough.

Sommaire : Une méthode de fusion sous vide, qui permet de déceler des traces d'éléments gazeux dans de très petits échantillons de métal, est décrite.

J. N. Gregory D. Mapper & J. A. Woodward *Analyst* 78, July 1953 414-427

Determination of Hydrogen in Wrought Aluminium Alloys — 173/III

See Abstract No. : 251/I

38 — DISTILLATION — 38

A Vacuum Anemometer — 174/III

See Abstract No. : 170/II

Molecular Distillation — 175/III

United Kingdom. The typical features of molecular distillation are : Reduced pressure of the atmosphere in the distillation chamber and a comparatively short distance between the evaporating and the condensing surface. If the latter is kept at 2 cm. and the pressure in the chamber at about 3×10^{-3} mm. Hg the distance between the 2 surfaces will be about equal to the mean free path of the gas molecules and vapours rising from the evaporation surface will reach the condensing surface without colliding with molecules of the residual gas. In an arrangement like this the rate of distillation is faster and the temperature at which distillation can be carried is lower than in the case of the conventional distillation procedure. The condenser surface is normally held at 20°C and this limits the type of materials which can be processed in this manner, as too volatile a material may re-evaporate from the condenser. On the other hand, substances of too high a molecular weight

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may decompose before reaching the temperature required for the distillation process. A batch molecular still and a continuous molecular still are described and shown in illustrations. The former consists of an evaporation tray which is heated by an element in the false bottom of the tray and is placed in a tube of small diameter carrying a jacket for water cooling. The tray is in a horizontal position, and the tube which serves as a condenser in a position slightly inclined to the horizontal, so that the condensate can collect at the bottom and be dispensed into a collecting vessel which in the particular case is fitted with 6 small receptacles to be filled separately. The continuous still consists of 2 de-gassers and 4 still stages which are built as separate units and assembled above each other on a vertical axis. The material to be processed is fed to the top unit and passes successively through all units until it reaches the bottom unit which is the hottest. Each unit is of the falling-film column type with the condenser surrounding the evaporator in the centre of the unit. Each unit is evacuated by a separate oil diffusion pump. The heat required is conveyed to the evaporator by radiation heaters. A special form of molecular still is the centrifugal still where the material to be evaporated is spread in a film across a rotating disc which has the added advantage of a reduced heating period and exceptional thinness of the liquid film. Where fractionation of the distillate is required, more complicated still designs have to be used. By way of example, details are given of a fractionating molecular still which consists of 3 troughs, placed at different heights adjacent to each other on a base plate which can be heated. Above the troughs are sloped condensing surfaces in a roof-like arrangement and the liquid condensed on each slope drips into the next higher trough. As a result the more volatile components of the processed material will accumulate in the top trough and the less volatile components in the bottom trough. Molecular distillation was first applied in 1922 by Bronsted and Hevesy for the separation of some of the isotopes of mercury. In 1928 Burch processed petroleum derivatives by this means and obtained various grades of Apiezon oils and greases which in turn led to the development of the oil diffusion pump. The centrifugal molecular still has been used for the concentration of vitamin A from fish liver oils.

Article by G. Burrows *Metro-Vick Gaz.* **25,** Aug. 1953 40-43

Sommaire : Un bref aperçu sur la distillation moléculaire avec référence particulière sur les dernières réalisations des distillateurs moléculaires.

Vacuum Distillation

176/III

Holland. As in fractional distillation in moderately high vacuum in conventional stills, such as bubble columns or packed towers, wherein the vapours and the liquid are brought into contact counter-currently, there is always an excessive pressure drop between the ends of the distillation column. According to this patent, the liquid is flowing through a series of pools and the vapour is flowing counter-currently while impinging against the pools to agitate the liquid. Thus the kinetic energy of the vapours is utilised, and the pressure drop caused by friction is considerably less than in the conventional columns. If a substantially horizontal column is used, this results in a minimal elevational difference between the first and the last pool, so that the pressure drop is reduced further. Structural details of substantially horizontal and of vertical columns are given.

(*Chemical Abstracts*)

N. V. de Bataafsche Petroleum Maatschappij *Dutch Pat.* 71,688

Sommaire : Une modèle spéciale d'une colonne de distillation qui tend à une réduction de lachute de pression entre les deux extrémités de la colonne.

Metal Column for Distillation of Corrosive Gas Mixtures at Low Temperatures

177/III

See Abstract No. : 236/I

A Variable—Temperature Vapour-Jacket for Fractionating Columns

178/III

United Kingdom. Normally electrical equipment is used to maintain adiabatic conditions required for precision separation in the operation of fractionating columns. However, electrical equipment in this application is affected by variations in mains voltage, changes in air temperature, etc. A vapour-jacket has been designed, shown in a sketch, which is to replace electrical equipment for the purpose. The vapour-jacket is part of a closed system consisting of a boiler containing pure methyl benzoate, air inlet valve and a vacuum pump. Its operation is based on the fact that at a fixed pressure the temperature of a vapour in equilibrium with a boiling liquid is constant. The boiler of the system is heated electrically and the pressure in the system is adjusted to suit, either by evacuation or by admittance of air as the case may be. In this manner, constant temperatures can be obtained in the system ranging from 45 to 193°C. by adjusting the pressure to any value between 0.1 and 760 mm. Hg. The temperature can be adjusted with a precision of $\pm \frac{1}{4}$°C.

Note by J. F. A. Williams *J. Sci. Instrum.* **30,** Sept. 1953 339

Sommaire : Une chemise de vapour sous forme d'un anneau de verre a été développée pour assurer le maintient des conditions adiabatiques dans une colonne de distillation.

Improvements r.t. the Degassing and Devolatilising Low Vapour Pressure Liquids

179/III

United Kingdom. Low vapour pressure liquids when subjected to high vacuum distillation tend to foam in the process which may lead to the distillate becoming contaminated with distilland which is carried over. This is due to excessive gas evolution and rapid evaporation of highly volatile components in the early stages of the operation. In order to eliminate foaming it is necessary to degas and devolatilise the material before it is fed to the distillation apparatus. The design of a degasser is described which is to be used for this purpose. It consists of an open-ended tube, positioned vertically, the lower end of which is connected by suitable means to a receptacle which contains the material to be degassed. This assembly is placed in the centre of a cylindrical container which is filled at the bottom with a suitable heating medium. An electrical heater is fitted to the

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bottom of the outer container and the whole assembly can be evacuated by means of a diffusion pump unit *via* a cold trap. The upper end of the latter tube is surrounded by a helical coil carrying cooling water. A suitable material for the aforementioned heating medium is Apiezon grease which liquefies at about 50°C and which has a vapour pressure as low as 10^{-3} mm. Hg at a temperature of 200°C. In operation the heating medium is brought to the temperature required for treatment of the liquid in question, as a result of which the gas occluded in the liquid and the more volatile components of that liquid will be driven up the tube and through its upper end into the outer vessel. Finally these materials reach the cold trap where they will be condensed. Due to the cooling action of the coil at the upper end of the inner tube a reflux action is initiated within the tube, returning the lesser volatile components to the receptacle. The precise degree of separation is determined generally by the heat applied *via* the heating medium in the outer vessel and the extent of cooling obtained at the upper end of the tube.

Sommaire : Une méthode de dégazage et de dévolatilisation de liquide à basse tension de vapeur avant le procédé actuel de distillation, est décrite.

G. Burrows & Metropolitan-Vickers Electrical Co. Ltd. *Brit. Pat.* 695,560

Additive for Jet Combustion Fuel — 180/III

See Abstract No. : 187/III

39 — MISCELLANEOUS PROCESSES — 39

An Inspection Method for Luminescent Screens — 181/III

See Abstract No: 211/I

Polythene Windows for Vacuum Systems — 182/III

United Kingdom. A method is briefly described for the manufacture of windows from polythene tape which can serve as X-ray tube windows giving a remarkably wide distribution of X-ray radiation, very desirable in certain special investigations.

Sommaire : Une méthode simple de fabrication de fenêtres, faites à partir d'un ruban de polythene, pour employer avec des tubes à rayons-X est donnée.

Letter by J. Hodkinson *Vacuum* 3, Oct. 1953 414-415

On the Problem of New Methods of Heat Insulation of Very Low Temperature Apparatus — 183/III

Russia. The creation of a moderate vacuum in an insulating space filled with a porous plastic material of low heat conductance coefficient was found to be an effective method of thermal insulation. The effectiveness is attributed to the fact that the pressure at which the molecular mean free path becomes equal to the wall-to-wall distance can be much higher if small pores rather than large spaces are present. Tests are reported in which a solidified foam of formaldehyde-uric tar was used. Trial with a hydrogen liquefier confirmed the suitability and expedience of the proposed method of insulation.

(*Nuclear Science Abstracts*)

Sommaire : La création d'un vide moderè dans un espace isolant rempli de matière plastique poreuse à basse conductibilité de chaleur donne une isolation effective de la chaleur.

Article by A. B. Fradkov *Doklady Akad. Nauk. S.S.S.R.* 81, 1951 549-551 also *A.E.C. Rep. No.* AEC-TR-2043 1953

Corrosion-Free Vacuum Filtration — 184/III

United States. Filtration of liquids likely to corrode ordinary materials will not affect the filter body, if it is made from ceramics. Ceramics recommended as construction material are : Standard stoneware, heat-shock resistant stoneware and porcelain. The first can be used at operational temperatures up to 130°F, the second at temperatures up to 200°F and porcelain up to 250°F. All three materials are completely impermeable to air and filters made from these materials can be used for vacuum filtration. Normally the filter has a cylindrical shape consisting of an upper chamber, a perforated support plate and a lower chamber. The vacuum connection is at the top of the lower chamber. A three-piece construction is preferable to a two-piece design where the perforated support plate is part of the upper chamber, in order to limit the loss in case of breakages. The medium retaining the solids which form the cake in the upper chamber, is usually cloth, placed on the perforated support plate. The weaving of the cloth must be open enough to avoid excessive plugging and tight enough to minimise the loss of fine particals. Instead of the cloth and the perforated support plate, a porous ceramic Filterstone plate can be used which is available in course, medium and fine grades and facilitates faster filtering rates. A faster filtering rate can also be obtained by increasing the pressure differential between the two chambers. But this is beneficial only in operations concerned with granular and crystalline solids. Where the cake is compressible, vacuum filters operated at about 10 p.s.i.a., have definite advantages. Some vacuum

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filters are exhausted by water jets but normally mechanical pumps are employed. A table, partly reproduced below, gives the pump capacities and power requirements for different sizes of ceramic filters. It is advisable

Size Filter		*Capacity*	*H.P. Motor*	*Speed of*
Upper Chamber	*Lower Chamber*	*c.f./min.*	*Required*	*Motor*
1 Gal.	2 Gals.	1	¼	1750
10	10	3½	¼	1750
50	50	6	½	1750
50	100	9	½–¾	1750
100	100	9	½–¾	1750

to use grease on the joints to stop the leakage of air and to insert a trap made from ceramic material between filter and pump. Ceramic vacuum filters are used in the pharmaceutical industry and by refiners of precious metals. They are not suitable for the treatment of soluble fluorides.

Sommaire : Des modèles efficients des filtres à vide en céramique sont décrit.

Article by W. Perkins & R. O. Weiss *Drug. & Cosm. Ind.* July 1953 44-46 & 129-133

O **Vacuum Pasteurisation**

185/III

United States. The control of flavour defects in dairy products is discussed. A vacuum pasteuriser, the Vacreator, still in the experimental stage, is described. It is a continuously working high-temperature direct steam unit. The processing time in the Vacreator is reduced to approximately 7 seconds compared with 2 minutes in the conventional short-time method and 1 hour in the batch system. Pasteurisation is effected at 194°F which is about 50°F higher than stipulated in the Law and the introduction of the apparatus will require a change of the Law. Preferably the Vacreator should be operated in conjunction with a regenerating plant. The processing operation on homogenised milk with 40°F raw milk is given. The raw milk is fed into the regenerator which it leaves at 104°F. It is then heated to 130°F in a pre-heater and enters the first of the three chambers of the Vacreator. Here it comes into direct contact with steam at 194°F. The second and the third chamber are kept at 165°F and 120°F respectively. The operational temperatures are controlled by varying the pressure in the chambers. The material discharged from the Vacreator is passed through a homogeniser and pumped through the pasteurised milk side of the regenerator cooling it to 56°F. Final cooling down to the initial temperature of 40°F is effected in a separate cooler. Compared with conventional apparatus heating costs are higher but cooling costs are lower. The efficiency of pasteurisation was determined by inoculating raw milk before treatment in the Vacreator with a culture of Freudenreichii M 25 at a rate of 4,500,000 per c.c. The results are given in a table, partly reproduced below.

Raw	4,500.000 p.c.c.
175°F	60,000
185°	28,000
195°	6,000

Sommaire : Une méthode spéciale de pasteurisation sous vide de produits laitiers est décrite.

Article by C. E. McIntire *Milk Plant Mthly.* **42**, May 1953 39-41

O **Continuous Soap Manufacture According to the Mazzoni Method**

186/III

Italy. The Mazzoni process for the production of solid soap facilitates soap making by a continuous method resulting in a considerable reduction of operating expenses. All conventional operations following the boiling of the soap are affected. The most important improvement is the introduction of the process of drying the soap after boiling in a large evacuated drum, into which the liquid soap, heated to 150°C, is fed through a nozzle. The soap is sprayed against the walls of the drum where it dries and cools down immediately. The dry soap is scraped off the walls in the form of flakes by rotating knives and is subsequently compressed in an extrusion press and passed through a cooling tunnel for final solidification. Due to processing in vacuum, oxidation of the soap ingredients can be prevented and crystallisation is under proper control, facilitating the manufacture of a completely transparent soap without having to add materials which may cause skin irritation in certain instances. Further, the coconut-oil content of the original mixture can be reduced from 30% to 5% without affecting the lathering properties of the soap. There are some 40 Mazzoni plants in operation already. In one fully mechanised factory in Italy three Mazzoni plants produce 140 tons of soap daily. Where 14 operatives were employed in the past, only 3 are now required.

Sommaire : Des détails sont donnés sur la production continue de savon, basée sur le procédé sous vide proposé par Mazzoni.

Article by M. Fock *Seifen, Oele Fette Wachse* **79**, 5.8.1953 414-416 see also G. Mazzoni & C. Mazzoni *Ital. Pat.* 478,301

Additive for Jet Combustion Fuel

187/III

United States. An additive for improving burning velocity of jet fuels may be prepared by heating sulphur vapour with various C_4 hydrocarbons or mixtures thereof (*e.g.* n–C_4H_{10}, n–C_4H_8 and butadienes) to 450–760° and immediately cooling to obtain thiophene tar. The tar is vacuum distilled at 100–200° and less than 10 mm. Hg to yield a red oil (45% yield) which is vacuum fractionated at 2 mm. Hg. The fraction boiling in the range

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120–125° is the desired additive. This product (15% of the original tar) has the molecular formula $C_4H_4S_3$. The addition of 0.5% of the product to 2,2,4–trimethylpentane increases the laminar burning velocity (with air) from 1.45 ft./sec. to 1.50 ft./sec. The addition of more than 2% does not seem to be beneficial.

Sommaire : Un procédé d'addition à'un produit améliorant la combustion des essences d'avions à réaction, et qui inclu l'emploi du vide, est décrit.

J. M. Godsey & Socony-Vacuum Oil Co. Inc. *U.S. Pat.* 2,645,569

Improvements r.t. the Degassing and Devolatilising of Low Vapour Pressure Liquids 188/III

See Abstract No. : 179/III

Evaporation 189/III

See Abstract No. : 159/III

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40 — GASES AND VAPOURS — 40

Fundamental Aspects of Combustion—Some Reactions of Atomic Hydrogen in Flames 61/IV
See Abstract No.: 234/I

A New Type of Helium Liquefier 62/IV
See Abstract No.: 172/II

Has the Physics of Very Low Temperatures any Significance in Practical Work ? 63/IV

United Kingdom. Various aspects of the physics of low temperatures are reviewed. In this connection the possibilities of producing very high vacua by means of low temperatures are briefly mentioned. At liquid helium temperatures all substances with the exception of helium itself have negligible small vapour pressures and therefore a helium bath can serve as a pumping means. The same applies to liquid hydrogen in connection with all gases except helium, neon, and hydrogen itself. Even helium could be pumped by applying such a technique if one chooses to operate in the temperature range of 1°K which can now be reached by the recently developed magnetic technique. At 0.1°K the vapour pressure of helium is reduced to 10^{-31} mm. Hg and at 0.03°K the vapour pressure is considerably lower still. The larger the low temperature system, the smaller is the comparative amount of heat entering into it. Thus, it is much easier to maintain a high vacuum at low temperature even in the case of large receptacles than it is in the case of room or elevated temperatures.

Sommaire : A la température de l'helium liquide toutes les substances, à l'exception de l'helium lui-même ont une tension de vapeur negligeable, donc, un bain d'helium liquide pourrait servir comme moyen de pompage.

Article by F. Simon *Kaeltetechnik* 5, Sept. 1953 246

Methods and Techniques for the Determination of Specific Surface by Gas Adsorption 64/IV

United Kingdom. A critical review is made of experimental procedures for the determination of specific surface and relative surface areas by the method of measuring the volume of gas absorbed on the surface of the solid. Volumetric and gravimetric techniques are described and compared and their suitability for routine and research purposes is indicated. The difficulties usually associated with the determination of gas adsorption isotherms are discussed, and a brief description is given of the methods used for calculating surface areas from the adsorption data.

(*Author*)

Sommaire : Une révue critique des méthodes expérimentales pour déterminer les surfaces spécifiques et relatives par la méthode de measurement du volume absorbé sur la surface de solide, est faite.

Article by A. S. Joy *Vacuum* 3, July 1953 254-278

A Vacuum Microbalance for Measuring Sorption in Dielectrics 65/IV
See Abstract No.: 153/II

41 — FREEZE DRYING — 41

Theory of the Vacuum Drying of Frozen Tissues 66/IV
See Abstract No.: 203/I

Freeze Drying and the Tissue Bank 67/IV
See Abstract No.: 243/I

The Frozen Bone Bank of the Netherlands Red Cross 68/IV

Holland. After Judet had demonstrated that calf-bone was suitable for use as heterograft material in humans, Meiss introduced this procedure in Holland. This led to the formation of a frozen bone bank (Bevroren Bot Centrale) run by the Netherlands Red Cross. The bank started operations in January 1952. Details of the processing of graft material are given. Preservation is effected by deep freezing. The financial aspect of the operations of the frozen bone bank are also considered.

Sommaire : Note sur les méthodes de traitements employés par la Banque des Os de la Croix Rouge Hollandaise.

Note by G. G. A. Mastenbroek (rec'd 14.9.53)

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A New Method of Preserving Fresh Human Milk

69/IV

Germany. Various methods of preparing human milk for preservation have been developed. Sterilising the milk for this purpose by boiling is still widely practised. It has been proved by Mayerhofer, Catal and other scientists that the process of boiling the milk is harmful to its quality. Vital ingredients such as Dold's and Weigmann's Inhibines, Lipase, vitamin C, etc. are destroyed. The freeze drying technique offers a means of preparing the milk for preservation in the raw state conserving its biological qualities. The basic elements of the procedure are described. The superior quality of human milk treated by the freeze drying technique has been proved in tests, the results of which are indicated in a table, partly reproduced below.

100 *c.c. Human Milk Contains* :—

	Raw	*Heated 10 Min. to 100°C*	*Freeze-Dried*
1. Vitamin A (International Units)	167 151 170	235 175 160	104 110 141
Average	169	210	118
2. Carotin	40.2 111.0 44.5	61.6 — 39	25 93 47
Average	72.5	67.5	55
3. Vitamin C (mg.)	4.48 4.48	3.5 4.4	4.6 4.3
Average	4.48	3.96	4.46
4. Vitamin B_2 (Lactoflavin)	54	62	56
5. Vitamin B_{12}	0.0305	0.0226	0.0263
6. Amylase (diastatic strength)	250	200	500
7. Catalase (number of catalases)	66	0	66
8. Lipase (lipase units)	22	0	17.6

Article by W. Catel, J. Pendl & O. Schiff *Dtsch. Med.Wschr.* **78**, Aug. 1953 1137-1140

Sommaire : Les avantages de la conservation du lait humain par lyophilisation sont donnée.

The Preservation of the Virus of Tomato Spotted Wilt in Dried Plant Material

70/IV

Australia. Some viruses lose their infectivity in *vitro* too quickly to permit proper investigation. The tomato spotted wilt virus belongs to this group. Its *in vitro* life in undiluted expressed juice at room temperature is limited to four hours. Best and Samuel succeeded in keeping the virus infective in solution for 35 days. Deep freezing freshly harvested material had some success. Only the application of the freeze drying method gave truly satisfactory results. Tomato leaflets infected with strain ' A ' and tissue of *N. glutinosa* infected with strain ' E ' were harvested and frozen at –20°C. After 2 hours the leaves were removed, cut into small pieces and freeze-dried at –30°C. After 8 hours a charge of 11 g. of the material was completely dry. The material was then stored at a temperature of –20°C. Samples withdrawn from storage after 125 days, suspended in a special solution and used for the innoculation of Tomato, Tobacco and *N. glutinosa* plants proved to be still infective although the mild strain ' E ' did not appear to be as well preserved as the strain ' A '.

Letter by R. J. Best & H. P. C. Gallus *Nature*, **172**, 15.8.1953 315

Sommaire : La lyophilisation s'est averée efficace comme moyen facilitant la conservation des virus " Tomate Spotted Wilt ".

Comparisons of the Survivals of Aspergillus Niger Spores Lyophilised by Various Methods

71/IV

United States. Extensive research has been carried out on the use of the freeze drying method for the preservation of bacteria and viruses, but little attention has so far been given to the preservation of mold cultures by this method. The authors report methodic experiments investigating the comparative advantages of three different freeze drying methods for the preservation of this type of material. *Aspergillus Niger* PRL 24 in beef serum has been used throughout these experiments. The three methods investigated were : (1) The so-called NRRL method developed by Rapier and Alexander, (2) the so-called PRL method and (3) the centrifugal freeze drying technique. The first essentially consists of snap-freezing the spores suspended in a suitable vehicle, evacuating the receptacle containing the suspension at low temperature and then at room temperature, and sealing off the tubes containing the dry material under vacuum. The second is similar to the first but the suspension is frozen in a freezing cabinet and primary drying is carried out in a vacuum desiccator. Then the vacuum is broken and secondary drying and sealing off is carried out in a suitable manifold arrangement. The process of the centrifugal freeze drying technique was based on the procedure developed by Greaves. The pressure during primary drying ranged from 100–50 μ Hg and during secondary drying from 50–25 μ Hg.

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Snap-freezing was carried out in the range of –35 to –65°C. Details of the method for the determination of the survival rate are given. A bench apparatus designed to be used for method (1) or (2) alternatively is described and shown in an illustration. The experiments were extended to study the effect of wetting agents such as ' Vel ' and ' Dreft ' and the effects of preliminary evaporation from the liquid state before freezing and drying took place. The results permit the following conclusions : The centrifugal freeze drying method appears to be superior to the other two methods investigated. A decided improvement in the survival rate was obtained by applying preliminary evaporation and ditto adding about 20% glucose to the processed material.

Sommaire : Trois méthodes de lyophilisation pour la conservation des spores d ' aspergillus niger ' sont comparées en fonction de leur efficacité.

Article by R. Haskins & J. Anastasiou *Mycologia* **45**, 1953 523-532

Preparation of Dry Clay-Gels by Freeze Drying 72/IV

United Kingdom. The author refers to the method applied by Hofman for the preparation of dry clay-gels by a freeze drying technique and reports on similar experiments carried out by him preparing samples of montmorillonite, the chief ingredient of bentonite, in 20–30 g. batches. The clay in aqueous solution was purified, saturated with the required base and frozen in the form of a layer covering the inner surface of a 2-litre flask. The flask was then evacuated and the moisture evaporated. The residual product had a bulk density of 0.12 gram per cm^3. The nitrogen area of calcium montmorillonite, determined by the Brunauer-Emmett-Teller method was 70 m^2/g. Degassing the gel at 10^{-5} mm. Hg for 100 hours caused it to break up into fine powder. A similar effect was obtained when heating it at 110°C for a prolonged period.

Sommaire : Une méthode de préparation de la pâte d'argile au moyen de la lyophilisation est décrite.

Letter by F. Call *Nature* **172**, 8.7.55 126

45 — ELECTRONICS — 45

The Importance of High Vacua to High Energy Nuclear Physics 73/IV

United Kingdom. A brief historical account is given of the development of cyclic accelerators, and present and future accelerators are discussed. An attempt is made to show the important place which these machines occupy in the most fundamental field of physics. The dependence of both, the machines and the physics, upon technology and in particular high vacuum technique is emphasised.

(*Author*)

Sommaire : Une brève révue historique du développement des accelerateurs cycliques est donnée et des accelerateurs présents et futures sont discutés.

Article by L. Riddiford *Vacuum* **3**, July 1953 231-244

The Design and Operation of a 4.5 MeV Microtron 74/IV

United Kingdom. The microtron described was similar to that previously constructed by Redhead *et al.* Electrons were injected by field emission in a microwave-resonant cavity and the radio-frequency and the steady magnetic field were adjusted so that the electrons were accelerated in a series of orbits distinctly separated from each other. A special permanent magnet provided a field of about 1,000 Gauss. The resonant frequency was of the order of 3,000 Mc/s transmitted by a wave-guide system from a pulsed magnetron source of 500 kW peak power. Pulse duration was 2 μ sec. with a repetition frequency of 200 c/s. The vacuum tank was made of brass, 21 inch. diameter and $3\frac{3}{4}$ inch. high, exhausted by a 4 inch. silicone oil diffusion pump. A pressure of 10^{-4} mm. Hg was found to be sufficient for reliable operation. Mean circulating currents of 0.2 μ A were obtained. An advantage of the new microtron was the large spacing of the orbits, (3.18 cm. in this case). The beam was extracted through a mild steel tube, the axis of which intercepted the orbit tangentially. The region inside the tube was field-free so that 50 % of the circulating current of the 9th orbit (at about 30 cm. diameter) could be brought into the laboratory through an 0.004 inch aluminium window. The divergence of the beam was 1.5° in the horizontal, and 0.3° in the vertical plane. The energy of the particles was calculated at 4.5 MeV with a spread of $\pm$0.07 MeV and was found to agree with calculations. It was concluded that about 2 Megawatt R.F. power was required for a microtron to compete with linear accelerators and this is in the process of being explored. The microtron's advantage lies in the simplicity of the construction, simpler magnet and the fact that the energy of the beam obtained is known and of a constant value. In view of the method of field emission injection of electrons, the output is unexpectedly stable.

Sommaire : Un microtron semblable à celui développé par Redhead est décrit en détail.

Article by C. Henderson F. F. Heymann & R. E. Jennings *Proc. Phys. Soc.* **66B**, August 1953 654-664

[The Cosmotron] Vacuum System. Part I—Vacuum Chamber 75/IV

United States. The design and assembly procedure of the vacuum chamber of the Brookhaven Cosmotron is described. In its final form the chamber consists of 4 quadrant sections and 4 straight sections, the latter designed to suit different special equipment in each case. The sections are joined to each other by bellows expansion units. The volume of the chamber as a whole is approximately 300 cu. ft. and the cross-section dimensions are : About 25 inch. in the horizontal direction and about $6\frac{1}{4}$ inch. in the vertical direction. Initial

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experiments with porcelain as construction material failed because it collapsed after evacuation. Similar experiments with plastics were abandoned for reasons of excessive outgassing. Solid stainless steel of low permeability was the material finally chosen. The side walls were made from continuous sheet, 1 inch thick, welded together from parts 15 ft. long, to give the required length and subsequently cold-rolled to the desired radii. The top and bottom of the straight sections, nearly 11 ft. long each and passing essentially through field-free regions, consisted also of sheet material but the top and bottom of the curved sections consisted of a ' grid ' of 2-inch. wide bars, spaced 1/16th inch apart, in order to avoid serious eddy-current effects. 608 bars were required for each quadrant section. All mechanical details of the construction are given. The bars had a thickness of 23/32 inch in the centre and were tapered at both ends. After evacuation the bars deflected by 3/16 inch. As the ends of each section had to be rigid the eight bars nearest to each end were joined together by means of steel pins in order to avoid too large a relative displacement between these and the rest of the bars after evacuation. A large relative displacement would badly stress the membrane placed over the bars and kept in position by hold-down strips to make the chamber vacuum-tight. The material to be used for these vacuum membranes was the subject of an extensive investigation. The metal Inconel was studied because of its high resistivity and its non-ferromagnetic properties. Welding of Inconel, however, presented major difficulties. The plastic Kel-F, a trifluorochloroethylene polymer had a satisfactory low vapour pressure and outgassing rate but it proved difficult to make an air-tight seal between two parts, necessary to form a continuous sheet of adequate length. It can be successfully moulded, however, and has therefore been used for the electrical insulation of bolts, screws and nuts. The synthetic rubber, Myvaseal, was finally chosen. It has the same vapour pressure and outgassing properties as Kel-F and, in addition, is more elastic and can easily be made into a continuous sheet covering the whole length of a quadrant section. Its electrical resistance properties are such that grounding of the grid bars by special wire, contemplated originally, could be omitted. A total of 1,100 sq. ft. of material, 1/16 inch thick, was used in the form of vacuum membranes. The total area exposed between the grid bars is 68 sq. ft. The total area liable to outgas after evacuation is largely determined by the finish of the grid bars as can be seen from the results of small-scale tests, shown in tabular form, reproduced below. The article concludes with an account of the procedures employed in assembling the vacuum chamber. (See Abstract 76/IV.)

Material for Covering Membrane	*Estimated Ultimate Pressure in Final Chamber*
Myvaseal on Rough Grid Bars	12×10^{-6} mm. Hg
Myvaseal on Machined Grid Bars	10×10^{-6}
Myvaseal on Machined and Greased Grid Bars	4.6×10^{-6}
Myvaseal on Polished Grid Bars	5.4×10^{-6}

Article by D. D. Jacobus & I. J. Polk *Rev. Sci. Instrum.* **24**, Sept. 1953 821-831

Sommaire : Des détails sur le modèle et ensemble de la chambre à vide du cosmotron de Brookhaven sont donnés.

[The Cosmotron] Vacuum System. Part II—Pumping System

76/IV

United States. Full details are given of the pumping system of the Brookhaven Cosmotron. The volume to be evacuated is approximately 300 cu. ft. The operational pressure must not exceed 5×10^{-6} mm. Hg to avoid excessive gas scattering. Twelve 20 inch. diffusion pumps in 2 groups of six each are placed at equal intervals in a trench surrounding the magnet ring, and are individually connected to the vacuum chamber *via* a 16-inch. valve and a rectangular transition box. The latter has been provided to keep all pumping and associated equipment well away from the particle beam. Each group of diffusion pump units is served by a common mechanical fore vacuum pump, rated at 300 c.f.m. of standard air, *via* a manifold. The diffusion pumps are connected to the manifold by a 3-inch. valve and the mechanical pump by a 6-inch. valve. A second mechanical pump, allocated to each group, serves as a stand-by but is normally used for rough pumping of the chamber. The diffusion pumps are operated with Litton-C oil and are of the 3-stage fractionating type (modified after delivery as single-stage pumps, rated at 7,000 l./sec.). A specially designed ' optically-opaque ' baffle, shown in a sketch, is fitted to the mouth of each diffusion pump. The effect of the new baffle on the speed of the pumps in the operational pressure range is nearly the same as that of the conventional baffle. The baffle is kept at 0°F by a 1 h.p. refrigerating unit. Each diffusion pump is backed by an 8-inch. single-stage booster pump. The diffusion pump and all its associated equipment is mounted on a single metal frame. In the first tests the diffusion pumps were run with Myvane-20 oil. This was found to have too high a vapour pressure for the purpose. Octoil-S oil was then chosen. Running the pumps with that fluid it was noticed that the fore vacuum pressure deteriorated appreciably. Tests of the oil of the mechanical pump indicated the presence of decomposition products of the Octoil-S oil which the existing oil purification system was unable to remove. It was decided therefore to revert to a petroleum-base oil and, finally, Litton-C oil was chosen. The article concludes with a detailed description of the vacuum system controls. (See Abstract 75/IV.)

Article by D. A. Kassner & C. Lasky *Rev. Sci. Instrum.* **24**, Sept. 1953 832-835

Sommaire : Des détails sur le système de pompage du cosmotron de Brookhaven sont donnés.

Vacuum Vessel Such as a Thermionic Valve

77/IV

See Abstract No. : 83/IV

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Abstract No. and References

A Survey of Present Knowledge of Thermionic Emitters

78/IV

United Kingdom. The usefulness of a number of metals as practical thermionic emitters is discussed. The effect of adsorbed films is described and illustrated in connection with emitters with monatomic layers of thorium, caesium and barium. The emission from compounds, in particular semi-conductors, metallic borides, carbides and nitrides is then considered. The general properties of oxides as cathodes, in particular barium-strontium-oxide, are then discussed. Finally, the performance of thoria as a thermionic emitter is described. A long bibliography is given.

(*B.S.I.R.A. Bulletin*)

Sommaire : L'emploi de métaux variés comme matériaux de construction pour les valves d'émission est discutés·

Paper by D. A. Wright *Proc. I.E.E., III* 100, May 1953 125-142

Crystallographic Variations of Field Emission from Tungsten

79/IV

United States. Fowler and Nordheim proposed first a now generally accepted theory of field emission on the base of which Sommerfield and Bethe derived an expression for the field emission current as follows :—

$$J = 1.55 \times 10^{-6} F^2 \varphi^{-1} \exp[-6.86 \times 10^7 \varphi^{\frac{3}{2}} F^{-1} v(y)]$$

Where J is the current density, φ the surface work function, F the electrical field, $y = 3.62 \times 10^{-4} F^{\frac{1}{2}}/\varphi$ and v(y) is a slowly varying elliptic function of y. This equation has been verfied in some respects but the dependence of the current density on the work function is not yet proved. The author reports on experiments aiming at the determination of relative field-emission currents along the main crystallographic directions of tungsten in an endeavour to contribute to the clarification of this problem. A projection tube was used, the envelope of which was covered in part by a phosphorus layer placed on top of a conducting layer deposited on the inner glass surface and measurements were taken by photometering the light output along the various crystallographic directions by means of a photomultiplier tube. In order to safeguard against distortion of the results by contamination phenomena an extremely low pressure was required in the tube. At conventional high vacuum of 10^{-8} mm. Hg the pattern emitted by the point cathode changes so quickly due to contamination that it is not possible to obtain a pattern of clean tungsten for long enough a period to facilitate proper observations and an accurate judgement on the influence of the work function of the various crystallographic faces. Prior to the experiments the tube, fitted with two ion gauges, one of the type described by Alpert (see Abstract No. 191/I), was thoroughly outgassed by heating the metal parts to high temperatures by the induction method and bombarding the phosphorus with thermionic electrons. This was repeated as often as was necessary to obtain a stable pattern between two cycles of the heating procedure. At this stage the pressure was 10^{-11} mm. Hg. The tube was then sealed off and pumped for about one week with the help of the ionisation gauge, after which period a pressure of 10^{-12} mm. Hg was obtained. Details of the experimental procedure are given and the results obtained by the author appeared to confirm the relationship between the work function and emission current predicted by theory.

Sommaire : Un rapport sur les recherches entreprises pour la mesure des courants d'emission relatives le long des directions principales cristallographiques du tungstène.

Article by M. K. Wilkinson *J. Appl. Phys.* 24, Sept. 1953 1203-1209

The Breakdown Voltages of Gaseous Discharges in Transverse Magnetic Fields, for the Pressure Range 10 to 10^{-8} mm. Hg

80/IV

Austria. Gives a very detailed experimental and theoretical account of breakdown voltage measurements for discharges in (essentially) coaxial cylinder tubes with axial magnetic fields. The electrode arrangement is similar to that previously studied by Wehrli and more recently and thoroughly by Penning. Experimental data for various gases are shown and a detailed theoretical treatment of breakdown mechanisms which involves a study of the motion of the ionising electrons in the magnetic field is given.

(*Science Abstracts*)

Sommaire : Des détails sont donnés sur résultats théoriques et expérimentaux des mesures de la tension de rupture de décharges dans des tubes coaxiaux ayant un champ magnétique axial.

Article by R. Haefer *Acta Phys. Austriaca* 7, No. 1, 1953 52-90

Thin Layers Transmitting Short Ultra-Violet Rays

81/IV

See Abstract No. : 226/I

A Quick-Change Seal Opener

82/IV

See Abstract No. : 168/II

47 — GLASS PROCESSING — 47

Vacuum Vessel Such as a Thermionic Valve

83/IV

Germany. It has been suggested to make certain evacuated components such as thermionic valves, wholly or in part, from ceramic materials. It is particularly advantageous to make the part supporting the electrodes from ceramics and the envelope from glass. For reasons of their thermal properties it is preferable to use

IV — Special Subsidiary Subjects — IV
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ceramics of comparatively low density such as Whetstone for the purpose, as distinct from high-density ceramics such as porcelain. In order to make low-density ceramics vacuum-tight, their surface has to be glazed. The joining of glazed ceramics and glass is a delicate and slow operation because the heat has to be applied gradually to avoid cracks in either material. The danger of cracks originates mainly from the uneven distribution of heat in the coat of glaze. To minimise this danger and to speed up the operation it is proposed in this patent to restrict the maximum thickness of the coat of glaze to 0.5 mm. The design of a thermionic valve based on the proposed sealing method is shown in a sketch.

Sommaire : La céramique non-poreuse à été employé comme base portant des electodes d'une valve thermionique, l'ampoule étant en verre.

E. Loepp & C. Lorenz A.G. *Ger. Pat.* 876,123

Vacuum Tight Glass-to-Metal Seals for Use in Electrical Engineering

84/IV

Germany. This is a comprehensive survey of all existing types of glass-to-metal seals. There are two major groups of such seals : (1) The glass and the metal have widely different expansion coefficients, such as molybdenum and quartz glass. (2) The expansion coefficients of both materials match closely, such as the Kovar alloy and the Corning Glass 7052, which has a $T\kappa_{100}$ value of about 300°C. Manufacturing seals from the first group of materials requires great care with regard to the actual sealing procedure and with regard to the shape of the components to be sealed together. However, within a limited temperature range such seals can be quite effective. Kovar seals are suitable for operation in a wide temperature range as long as the maximum temperature of 200°C is not exceeded for any appreciable length of time during the use of the component. The Kovar material is an alloy consisting essentially of 54% iron, 29% nickel and 17% cobalt. Similar materials of a composition slightly varying from that of cobalt are sold under different trade names. Using these materials there is no limitation of size and shape of the seal to be manufactured. Various details of the sealing technique and of seal designs are discussed and shown in illustrations. Generally speaking, seals should be so designed that a minimum of tensile stress is exerted in the glass during operation, as the tensile strength of the glass is only 900 kg/cm² whereas the compressive strength is 9,000 kg/cm². It is good practice in the manufacture of such seals to allow for a slightly larger expansion tolerance in the Kovar component than in the glass component. The glass component of a Kovar seal, in a thermionic valve for instance, subjected to an excessive temperature for a prolonged period is liable to be destroyed by electrolytic action. An accelerated test has been devised which gives information on the potential useful service life of the seal under the operational conditions for which it is designed. Details of this test are given. The Corning Glass 7052 normally used for these seals has a high boric acid content but no lead is included. In order to increase insulation a lead containing Kovar glass has been developed in France under the name ' verre E ' which contains 28.7% lead oxide and has a $T\kappa_{100}$ value of 322°C. As long as high-frequency is used in the sealing process this glass shows good adhesion properties but adhesion is less satisfactory if the more conventional methods of heating are applied. The reduced adhesion is due to the fact that lead has a high electron affinity to iron and when the molten glass is coming into contact with the Kovar metal the lead separates out in the form of small lead droplets of a size varying from 2 to 0.3μ. Often, for instance in the case of transmitter valves incorporating Kovar seals, the low electrical conductivity of the metal results in an undue rise of temperature within the seal. This can be overcome by improving the conductivity of the Kovar metal component prior to sealing by coating it with a material such as copper, silver or gold. The thickness of this coating should not be less than 30 μ as otherwise the coating may diffuse into the glass during the sealing operation, defeating the purpose of the coating.

Sommaire : Général aperçu sur la soudure verre-métal.

Article by W. Duesing *Glastech. Berichte* **26**, August 1953 232-238

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Zemany, P. D.	Thermal Degradation of Polystyrene.	115/I
Zorll, U.	The Limit of Accuracy in the Measurement of the Thickness of a Thin Film Using the Koester Interference Double Prism.	104/I
Zwiebel, N., *et al.*	Carbon Dioxide.	111/II

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Abbreviations and Publishers of Journals Quoted in this Volume

Acta Cryst.	Acta Crystallographica, Ejnar Munksgaard, Copenhagen, Denmark.
Acta Phys. Austriaca	Acta Physica Austriaca, Springer Verlag, Mölkebastei 5, Vienna, 1, Austria.
Acta Metallurg.	Acta Metallurgica, 57, East 55th Street, New York 22, N.Y., U.S.A.
A.E.C. Reports	U.S. Atomic Energy Commission Reports,
A.E.C.D. Reports	Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
A.E.R.E. Reports	U.K. Atomic Energy Research Establishment Reports, Her Majesty's Stationery Office, P.O. Box 569, London S.E.1, England.
Amer. Ceramic Soc. Bull.	The American Ceramic Society Bulletin, 2525, North High Street, Columbus 2, Ohio, U.S.A.
Amer. Export Industr.	American Exporter Industrial, Johnston Export Publishing Co., 386, Fourth Avenue, New York 16, N.Y., U.S.A.
Amer. Foundryman	American Foundryman, American Foundrymen's Society, Inc., 616, St. Michigan Avenue, Chicago 5, U.S.A.
Analyst	The Analyst, The Society of Public Analysts and Other Analytical Chemists, 7-8, Idol Lane, London, E.C.3, England.
Ann. Phys.	Annalen der Physik, Johann & Ambrosius Barth, Salomonstr. 18b, Leipzig C.1, Germany.
Analyt. Chem.	Analytical Chemistry, American Chemical Society, 1155, Sixteenth Street, N.W., Washington 6, D.C., U.S.A.
Appl. Sci. Res.	Applied Scientific Research, Martinus Nijhoff, The Hague, Holland.
Ark. Fys.	Arkiv For Fysik, Kungliga Svenska Vetenskapsakademien, Stockholm, 50, Sweden.
Arch. Tech. Messen.	Archiv für Technisches Messen, R. Oldenbourg Verlag, Lotzbeckstrasse 2b, München, Germany.
Atomics	Leonard Hill Ltd., Stratford House, Eden Street, London N.W.1, England.
Aus Forschung und Produktion	Degussa, Deutche Gold- und Silber Scheideanstalt, Frankfurt/Main, Germany
Bild & Ton	Henschel Verlag Kunst und Gesellschaft, Oranienburger Strasse 67, Berlin N.4, Germany.
Biochim. Biophys. Acta	Biochimica et Biophysica Acta, Elsevier Publishing Co., Amsterdam, Holland.
Brit. Gel. & Glue R.A.	British Gelatine and Glue Research Association, 2a, Dalmeny Avenue, Holloway, London N.7, England.
Brit. J.Appl.Phys.	British Journal of Applied Physics, The Institute of Physics, 47, Belgrave Square, London S.W.1, England.
B.S.I.R.A. Bull.	British Scientific Instrument Research Association, Information Department, ' Sira ', Southill, Elmstead Woods, Chislehurst, Kent, England.
Bull. Astr.	Bulletin Astronomique, Librairie Gauthier-Villars, 55, Quai des Grands-Augustins, Paris, France.
Bull. Inst. Int. du Froid	Bulletin de l'Institut International du Froid, Institut International du Froid, 177, Boulevard Malesherbes, Paris 17e, France.
Bull. Math. Biophys.	Bulletin of Mathematical Biophysics, University of Chicago Press, 5750, Ellis Avenue, Chicago, U.S.A.
Canad. Chem. Process.	Canadian Chemical Processing, 137, Wellington Street West, Toronto, Ontario, Canada.
Canad. J. Chem.	Canadian Journal of Chemistry, National Research Council, Ottawa, Canada.
Canad. J. Phys.	Canadian Journal of Physics, The National Research Council, Ottawa, Canada.
Canad. J.Technol.	The Canadian Journal of Technology, The National Research Council, Ottawa, Canada.
Ceramic Age	Ceramic Publishing Co Inc., 421, Parker Street, Newark 4, N.J., U.S.A.
Chemical Abstracts	The American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Chem. Engng. Progr.	Chemical Engineering Progress, American Institute of Chemical Engineers, 120, East 41st Street, New York 17, N.Y., U.S.A.
Chem. & Industr.	Chemistry and Industry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Chem. Technik	Chemische Technik, VEB Verlag Technik, Unter Den Linden 12, Berlin, N.W.7, Germany.

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Chem. Process Engng.	Chemical and Process Engineering, Leonard Hill Ltd. 17, Stratford Place. London W.1, England.
Chem. Ing. Tech.	Chemie Ingenieur Technik, Chemie Verlag, G.m.b.H., Hauptstrasse 129, Weinheim/Bergstrasse, Germany.
Compr. Air Mag.	Compressed Air Magazine, Compressed Air Magazine Co., Phillipsburg, N.Y., U.S.A.
Conference Opt. Soc. Amer.	Conference of the Optical Society of America, c/o Journal of the Optical Society of America, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
C.R. Acad. Sci., Paris	Comptes Rendus des Séances de L'Academie des Sciences, Messrs. Gauthier-Villar, 55, Quai des Grands-Augustine, Paris, France.
C.R. Comm. 1 & 2 I.I.F., Louvain	Comptes Rendus Commissions 1 & 2, Institut International du Froid, c/o Bulletin de l'Institut International du Froid, 177, Boulevard Malesherbes, Paris 17e, France.
Design & Res. Centre Bull.	Design and Research Centre Bulletin, Design and Research Centre, Goldsmiths' Hall, Foster Lane, London E.C.2, England.
Dtsch. Med.Wschr.	Deutsche Medizinische Wochenschrift, Verlag Georg Thieme, Diemershalden Strasse 47, Stuttgart, E. Germany.
Drug. & Cosm. Ind.	Drug and Cosmetic Industry, Drug Markets Inc., 101, West 31st Street, New York 1, N.Y., U.S.A.
D.S.I.R. Bibliography (& Reports)	Bibliography of Industrial Drying, Department of Scientific and Industrial Research, Her Majesty's Stationery Office, P.O. Box 569, London S.E.1, England.
Electronics	McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 18, N.Y., U.S.A.
Endeavour	Imperial Chemical Industries Ltd., 26, Dover Street, London W.1, England.
Engineer	The Engineer, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London W.C.2, England.
Exp. Cell Res.	Experimental Cell Research, Academic Press Inc., 125, East 23rd Street, New York 10, N.Y., U.S.A.
Food Engng.	Food Engineering, McGraw-Hill Publishing Co. Inc. 330, West 42nd Street, New York 36, N.Y., U.S.A.
Giesserei	Giesserei Verlag G.m.b.H, Düsseldorf, Germany.
Glas-Email-Ker-Tech.	Glas-Email-Keramo-Technik, P.O. Box Verlag Brunke Garrels, Hamburg 26, Germany.
Glas & Hochvakuum Tech.	Glas und Hochvakuum Technik, Rudolf A. Lang Verlag, Leibnizstrasse 64, Berlin-Charlottenburg 4, Germany.
Glastech. Berichte	Glastechnische Berichte, Verlag der Deutschen Glastechnischen Gesellschaft, Untermainkai 12, Frankfurt (Main), Germany.
Helv. Phys. Acta	Helvetica Physica Acta, Physikalische Anstalt, Universität, Klinverbergstrasse 82, Basle, Switzerland.
I.E.E. Paper	Institution of Electrical Engineers Paper (Special Print), c/o Proceedings of the Institution of Electrical Engineers, Institution of Electrical Engineers, Savoy Place, London W.C.2, England.
Industr. Chem.	Industrial Chemist, St. Margaret's Technical Press, Ltd., 35, Tothill Street, London S.W.1, England.
Industr. Engng. Chem.	Industrial and Engineering Chemistry American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Industr. Finish.	Industrial Finishing, Arrow Press Ltd. 157, Hagden Lane, Watford Herts., England.
Instruments	Instrument Publishing Co. Inc., 921, Ridge Avenue, Pittsburgh 12, Pa., U.S.A.
Invest. Phys.	Investigationes Physicae, 40, Carlton Crescent, Summer Hill, Sydney, N.S.W., Australia.
Iron Age	Chilton Co. Inc., Chestnut and 56th Streets, Philadelphia 39, Pa., U.S.A.
Iron & Steel	The Louis Cassier Co., Ltd., Dorset House, Stamford Street, London S.E.1, England.
Jenaer Jahrbuch	Kommissionsverlag Gustav Fischer, Jena, Germany.
J. Amer. Ceramic Soc.	Journal of the American Ceramic Society, American Ceramic Society, 2525, N. High Street, Columbus 2, Ohio, U.S.A.
J. Amer. Oil Chem. Soc.	Journal of the American Oil Chemists Society, The American Oil Chemists' Society, 35, E. Wacker Drive, Chicago 1, Ill., U.S.A.
J. Appl. Phys.	Journal of Applied Physics, American Institute of Physics. 57, East 55th Street, New York 22, N.Y., U.S.A.

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J. Chem. Educ.	Journal of Chemical Education, Div. of Chemical Education of the American Chemical Society, Scripps Institution, La Jolla, California, U.S.A.
J. Chem. Phys.	The Journal of Chemical Physics, American Institute of Physics, Inc., 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Imp. College Chem. Engng. Soc.	Journal of the Imperial College Chemical Engineering Society. Imperial College Chemical Engineering Society, Editorial Board, Dept. Chemical Engineering, Imperial College, London S.W.7, England.
J. Inst. Fuel	Journal of the Institute of Fuel, Institute of Fuel, 18, Devonshire Street, Portland Place, London, W.1, England.
J. Inst. Metals	Journal of the Institute of Metals, The Institute of Metals (Inc.), 4, Grosvenor Gardens, London, S.W.1, England.
J. Iron Steel Inst.	Journal of the Iron and Steel Institute, Iron and Steel Institute, 4, Grosvenor Gardens, London S.W.1, England.
J. Med. Lab. Tech.	Journal of Medical Laboratory Technology, Institute of Medical Laboratory Technology, 9, Harley Street, London W.1, England.
J. Metals	Journal of Metals, American Institute of Mining and Metallurgical Engineers, Inc., 29, West 39th Street, New York 18, N.Y., U.S.A.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Phys. Radium	Journal de Physique et le Radium, La Société Francaise de Physique, 10, rue Vauquelin, Paris 5e, France.
J. Phys. Soc., Japan	Journal of the Physical Society of Japan, Physical Society of Japan, c/o Dept. Physics, Faculty of Science, Tokyo University, Bunkyo-ku, Tokyo, Japan.
J. Res. Nat. Bur. Stand.	Journal of Research of the National Bureau of Standards, U.S. Government Printing Office, Washington 25, D.C., U.S.A.
J. Sci. Food Agric.	Journal of the Science of Food and Agriculture, Society of Chemical Industry, 56, Victoria Street, London S.W.1, England.
J. Sci. Instrum.	Journal of Scientific Instruments, Institute of Physics, 47, Belgrave Square, London S.W.1, England.
Kältetechnik	Kältetechnik, Verlag C. F. Müller, Postfach, Karlsruhe, Germany.
J.S.M.P.T.E.	Journal of the Society of Motion Picture and Television Engineers, Society of Motion Picture and Television Engineers, 40, West 40th Street, New York 18, N.Y., U.S.A.
Lab. Practice	Laboratory Practice, United Trade Press Ltd., 9, Gough Square, Fleet Street, London E.C.4, England.
Light Metals	Temple Press Ltd., Bowling Green Lane, London E.C.1, England.
Machinist	McGraw-Hill Publishing Co., Ltd., 95, Farringdon Street, London E.C.4, England.
Mater. & Meth.	Materials and Methods, 330, West 42nd Street, New York 18, N.Y., U.S.A.
Meat Industr.	Meat Industry, Mezhdunarodnaja Kniga 32-34 Smolensk Square, Moscow 200, U.S.S.R.
Mech. World & Engng. Rec.	Mechanical World and Engineering Record, 31, King Street West, Manchester 3, England.
Mém. Soc. Roy. Belge. Industr.	Memoires de la Société Royale Belge des Ingénieurs et des Industriels. Hôtel Ravenstein, 3, rue Ravenstein, Bruxelles, Belgium.
Metal Finish.	Metal Finishing, Finishing Publications, Inc., 11, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Industr.	Metal Industry, Iliffe & Sons Ltd., Dorset House, Stamford Street, London, S.E.1, England.
Metal Progr.	Metal Progress, The American Society for Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.
Metal Treatm.	Metal Treatment, Industrial Newspapers Ltd., 49, Wellington Street, Strand, London W.C.2, England.
Metall	Metall Verlag Gm.b.H, Hubertusallee 18, Berlin—Grunewald, Germany.

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Contd.

Metallurgia	The Kennedy Press, Ltd., 31, King Street West, Manchester 3, England.
Metro- Vick. Gaz.	Metropolitan-Vickers Gazette, Metropolitan-Vickers Electrical Co. Ltd., Trafford Park, Manchester 17, England.
Milk Plant Mthly.	Milk Plant Monthly, National Milk Publishing Co., 327, S. La-Salle Street, Chicago, 4, U.S.A.
Mill & Factory	205, East 42nd Street, New York 17, N.Y., U.S.A.
Mod. Res. Techniques Phys. Metallurgy	Modern Research Techniques in Physical Metallurgy, Report on Conference held in October, 1952, American Society for Metals, Cleveland, Ohio, U.S.A.
Monatsh.	Monatshefte für Mathematik und Physik, Akademie der Wissenschaften, Vienna, Austria.
Mon. Not. Roy. Astr. Soc.	Monthly Notices of the Royal Astronomical Society, Royal Astronomical Society, Burlington House, London W.1, England.
Mycologia	Mycological Society of America, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
N.A.C.A. Rep.	National Advisory Committee for Aeronautics, Washington D.C., U.S.A.
Nature	Macmillan and Co., St. Martin's Street, London W.C.2, England.
Naturwiss.	Naturwissenschaften, Springer Verlag, Jebenstrasse 1, Berlin—Charlottenburg, Germany.
N.B.S. Report	National Bureau of Standards Report, Office of Scientific Publications, National Bureau of Standards, Washington 25, D.C., U.S.A.
Nederlands Tijdschr. Natuurk.	Nederlands Tijdschrift voor Natuurkunde, Martinus Nijhoff, Lange Voorhout No. 9, The Hague, Holland.
N. J. Cer. Res. Sta. Rep.	Technical Reports, N.J. Ceramic Research Station, Rutgers University, New Brunswick, N.J., U.S.A.
N.R.C. Canada Rep.	National Research Council of Canada Report, National Research Council of Canada, Ottawa, Canada.
Nuclear Science Abstr.	Nuclear Science Abstracts, U.S. Atomic Energy Commission, Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
Nucleonics	McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 18, N.Y., U.S.A.
Oak Ridge Nat. Lab. Reports	Oak Ridge National Laboratory Reports, Y-12 Area, Oak Ridge, Tenn., U.S.A.
Optik	Wissenschaftliche Verlagsgesellschaft m.b.H, Stuttgart, Germany.
Oyo Butsuri	Journal of Applied Physics, The Society of Applied Physics, Faculty of Technology, University of Tokio, Japan.
Paint, Oil, Colour J.	Paint, Oil and Colour Journal, Scott Greenwood & Son Ltd., 83-86, Farringdon Street, London E.C.4, England.
Paper Mkr.	The Paper-Maker and British Paper Trade Journal, S. C. Phillips & Co., Ltd., 3, Tudor Street, London E.C.4, England.
Periodic. Mathem.-Phys. Astron. (Zagreb)	Periodicum Mathematico-Physicum Astronomicum (Zagreb), Hrvatski Priodoslovno Drustvo, Marulicev trg. 19, Zagreb, Yugoslavia.
Petrol. Ref.	Petroleum Refiner, The Gulf Publishing Co., Box 2608, Houston 1, Texas, U.S.A.
Pharm. Acta Helv.	Pharmaceutica Acta Helvetiae, Schweitzerischer Apotheker-Verein, Sihil Strasse 37, Zürich, Switzerland.
Philips Tech. Rev.	Philips Technical Review, Philips Electrical Ltd., Century House, Shaftesbury Avenue, London W.C.2, England.
Phys. Rev.	Physical Review, American Institute of Physics, Brookhaven National Laboratory, Upton, Long Island, N.Y., U.S.A.
Plansee Proc.	Plansee Proceedings, Metallwerk Plansee G.m.b.H, Reutte/Tyrol, Austria.
Planseeber. Pulvermet.	Planseeberichte für Pulvermetallurgie, Metallwerk Plansee G.m.b.H., Reutte/Tyrol, Austria.
Plastics Progr.	Plastics Progress, Iliffe & Sons Ltd. Dorset House, Stamford Street, London S.E.1, England.
Plating	The American Electroplaters Society, American Building, 445, Broad Street, Newark 2, N.J., U.S.A.
Proc. Amer. Soc. Test. Mater.	Proceedings of the American Society for Testing Materials, The American Society for Testing Materials, 20th and Northampton Streets, Easton, Pa., U.S.A.

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Proc. I.E.E.	Proceedings of the Institution of Electrical Engineers, Institution of Electrical Engineers, Savoy Place, London W.C.2, England.
Proc. Phys. Soc.	Proceedings of the Physical Society, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London S.W.7, England.
Proc. Roy. Soc.	Proceedings of the Royal Society, The Royal Society , Burlington House, Piccadilly, London W.1, England.
Prod. Finish.	Product Finishing, Sawell Publication Ltd., 4, Ludgate Circus, London E.C.4, England.
R.C.A. Rev.	R.C.A. Review, R.C.A. Laboratories, Radio Corporation of America, Princeton, N.J., U.S.A.
Refrig. Engng.	Refrigerating Engineering, American Society of Refrigerating Engineers, 40 W. 40th Street, New York 18, N.Y., U.S.A.
Rep. Progr. Phys.	Reports on Progress in Physics, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London S.W.7, England.
Rev. Mod. Phys.	Review of Modern Physics, American Institute of Physics, Lancaster, Pa., U.S.A.
Rev. Prat. Froid	Revue Pratique du Froid, 254, Rue de Vaugirard, Paris 15e., France.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Science	American Association for the Advancement of Science, 1515, Massachusetts Avenue, N.W., Washington 5, D.C., U.S.A.
Science Abstracts	Institution of Electrical Engineers, Savoy Place, London W.C.2, England.
Sci. Light	Science of Light, Institute for Optical Research, Tokyo University of Education, 4-400, Hyatunin-Cho, Shinjuku-Ku, Tokyo, Japan.
Scient. Amer.	Scientific American, Scientific American, Inc., 2, West 45th Street, New York 36, N.Y., U.S.A.
Seifen, Oele Fette Wachse	Augsburg 2, Postfach, Germany.
S.P.E.J.	Society of Plastics Engineers Journal, Society of Plastics Engineers, Suite 116-118, 34 East Putnam Avenue, Greenwich, Conn., U.S.A.
Sprechsaal	Sprechsaal für Keramik Glas Email, Verlag Mueller and Schmidt, Coburg, Germany.
Stahl u. Eisen	Stahl und Eisen, Düsseldorf, Germany.
Structural Engr.	The Structural Engineer, Institution of Structural Engineers, 11, Upper Belgrave Street, London S.W.1, England.
Telef. Ztg.	Telefunken Zeitung, Technisch-Wissenschaftliche Mitteilungen, Telefunken Ges. für Drahtlose Telegraphie, 32-34, Nehringdamm, Berlin S.W.61, Germany.
Textile Industr.	Textile Industries, A.W.R.C. Smith, 806, Peachtree Street, N.E., Atlanta, Ga., U.S.A.
The Times (Industr. Suppl.)	' The Times ' Industrial Supplement. ' The Times ' Publishing Co. Ltd., Printing House Square, London E.C.4, England.
Trans. Faraday Soc.	Transactions of the Faraday Society, The Faraday Society, Aberdeen University Press Ltd., 6, Upper Kirkgate, Aberdeen, Scotland.
Trans. I. Chem. E.	Transactions of the Institution of Chemical Engineers. The Institution of Chemical Engineers, 56, Victoria Street, London S.W.1, England.
Univ. Calif. Rad. Lab. Rep.	University of California Radiation Laboratory Reports, University of California, Berkeley, California, U.S.A.
Ver. Koninkl. Vlaamse Acad. Wetensch.	Verhandelingen van de Koninklijke Vlaamse Academie voor Wetenschappen, Paleis der Academiën, Hertogelijke Straat, 1, Brussels, Belgium.
Le Vide	Le Vide, Société Française des Ingénieurs Techniciens du Vide, 44, Rue de Rennes, Paris 6e., France.
Weld. J.	Welding Journal, The American Welding Society, 33, West 39th Street, New York 18, N.Y., U.S.A.
World Sci. Rev.	World Science Review, Kenneth de Courcy, Alderbourne Manor, Gerrards Cross, Bucks., England.
Yale J.Biol.Med.	Yale Journal of Biology and Medicine, 333, Cedar Street, New Haven 11, Conn., U.S.A.
Zacchia	Rivista di Medicina Legale e delle Assicurazioni, Viale Dell 'Università, Roma, Italy.

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Z. Angew Phys.	Zeitschrift für Angewandte Physik, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.
Z. Phys.	Zeitschrift für Physik, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.
Z. V.D. I.	Zeitschrift des Vereines Deutscher Ingenieure, Prinz Georg Strasse 77/79 Düsseldorf, Germany.
Zh. Tekh. Fiz.	Zhurnal Tekhnicheskoi Fiziki, Akademiya Nawk S.S.S.R., Moscow, Russia.

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RESIN-SOLVENT COMPATIBILITY C

compiled by
S. BATESON

see Article on

ALUMINIUM REFLECTING FILMS APPLIED TO

Vacuum Vol. III, No. 1, January, 1953, 35-42a (published

1	2	3	4	5	6				7	8	9
	Chemical Type	*Surface Coating Materials*	*Film Formation*	*Solvents*	*Resistance Rating*				*Application Problems*	Aluminising	*Application F*
					Water R.T.	*Oils (Veg.)*	*Solvents–mild Handling–nor.*		*Undercoating Characteristics on Base Plastic*		*Overcoating Cha on Base C*
SYNTHETIC THERMOPLASTIC SURFACE COATINGS											
*	Cellulose Derivatives	Cellulose Nitrate	SR, R	E, K	F	F	P	G	Hard, brittle, air dry film, lacks adhesion		Crazing–solvent penetra
		Cellulose Acetate	SR, R	E, K	P	P	M	M	Mod., hard, air dry film, cobweb spray, do.		,, ,,
		Cellulose Acetate Butyrate	SR, R	E, K	F	M	M	M	,, ,, ,, ,, ,, ,, ,, ,,		,, ,,
		Ethyl Cellulose	SR, R	E, K	F	P	P	M	Tough, gloss film–air dry–fair adhesion		Top surface film plastici
	Acrylic Esters	Methyl Methacrylate ..	SR, R	CI, E, H	G	G	G	M	Brittle film–cobweb spray–lifts when dry		Crazing–solvent penetra
	Vinyl Resins	Styrene Resin	SR, R	E, H, K	E	E	M	G	Hard, brittle film–fair adhesion–lifts–aging		Crazing–solvent penetra
		Styrenated Alkyd or Oil ..	O, R,M	H, V	G	G	M	M	Partial polymer–fairly good adhesion, suitable as base coat sealer for Styrene		Not sufficiently mar resi solvent addition No.
		Vinyl Alcohol–	SR, R	A, W	P	G	G	G	Tough, gloss film–good adhesion		Poor water resistance–pl
		Vinyl Butyral–	SR, CH } R,M	A, E	G	F	G	E	Hard, tough film–poor adhesion		Plasticized and peels in c
		Alkyd Resins–Glycerol–Oil Combinations	SR, RM	E, H, W	G	G	M	G	Mod. hard film, too long drying for production		Not sufficiently mar resi Limited use with solven
SYNTHETIC THERMOSETTING SURFACE COATINGS											
	Phenol Res. Comb.	Phenol–Formaldehyde Resins (Pure)	P, H	A, K, W	E	E	E	E	Too high baking temp. for thermo-plastics		Hard, brittle film–lifts on
		Phenol-Formaldehyde Resins (Modified)	P, MH CH	A, H, E	E	E	G	E	Too high baking temp. for thermo-plastics		Durable, hard film–tend
●	Melamine Res. Comb.	Melamine–Formaldehyde Resins	P, H	A, H	E	E	E	E	Too high baking temp. for thermo-plastics, good adhesion		Very brittle film–age craz
		Melamine–Alkyd Resins, 1 : 1, 2 : 1	P, M	A, H 2 : 3	E	E	G	E	Some tendency to blush in high humidity		Durable, mar resistant fi
		Melamine–Alkyd Resins, 1 : 1, 2 : 1 (for Polystyrene)–	P, M	A (Butanol)	G	E	G	E	Revised formulation–no Xylol–non-drying Alkyd		Hard, durable film–requ heat
	Urea Resin Comb.	Urea–Formaldehyde Resins	P, H	A, H	G	E	E	E	Brittle film–high baking temp.–age crazing		Tendency towards britt lift'g.
		U–F Oil Resin Combinations	P, M	A, H	E	E	G	G	Some sacrifice in hardness, improved adhesion		Less checking, etc., infer Alkyd
		Urea Resin–Vinyl Butyral ..	P, M	A (Butanol)	E	E	G	E	Poor adhesion to base plastic–strips easily		Hard, durable film–requi Good adhesion, mar resi

KEY TO CODE

Code for Film Formation (Column 4)

*	SR—Solvent Removal—air or force dry	R—Cured at room temperatures, 70-80°F.	*
	CH—Chemical Hardening	M—Cured at moderate temperatures, 130-160°F.	●
	O—Oxidation in presence of metallic dryers	H—Cured at elevated temperatures, 275-310°F.	
●	P—Polymerization		

Code for Solvents

	A—Alcohols—higher fractions
	CI—Chlorinated Hydrocarbons
	E—Fatty Acid Esters
	H—Aromatic hydrocarbons

For the meaning of the symbols * and ● see text in the ad

O TO PLASTICS

ished August, 1954).

9	10								11
ation Problems	*Effectiveness of Surface Coating with*								
ng Characteristics Base Coating	*Acetate H* 1	*Butyrate MH* 2	*Butyrate H* 3	*Lucite* 4	*Melamine* 5	*Phenolic* 6	*Styrene* 7	*Urea* 8	*Trade Names*
		✱							
penetration to undercoat	As air dry protective spray coat–except 4&7								Berryloid 1 GP 31
" " "	" " " " " " " "							1&7	Berry Bros. 29 Clear
" " "	" " " " " " " "							2&3	
plasticized by hair oils	Primarily formulated for Polystyrene								Schwartz Rez-N-Lac
penetration to undercoat	As air dry, resistant top coat–except 4 & 7								
penetration to undercoat	Not widely used–lacks flexibility–wet prop.								
ar resistant–soft–critical n No. 1–not for No. 2	Formulated for Polystyrene base coating								Aulcraft LX.9011 (Styrenated Oil) No. 1 Aulcraft LX.9036 (Styrenated Oil) No. 2
nce–plasticised by oils	Inflexible–properties affected by moisture								
els in contact with hair oil	As force-dry top protective coating Primarily formulated for Polystyrene								Aulcraft { LX8813 { LX8894 Vinyl Butyral OL125 { OL129 Wax Free Shellac
ar resistant solvents	As base plastic coating–except 4 & 7								C. 1 L, GR. 1
lifts on aging–dark colour	Primarily formulated for No. 6 base coating								Plastics & Chem. Ltd., S–1346–H
n–tendency to lift–ditto	As base coating for thermosetting plastics								Aulcraft VX 8961
ge crazing–requires heat	As base or top coating for Thermosets 5, 6 & 8								Plaskon 3375 to 3399
stant film–catalyst reqd.	As base and top coating–force dry–for Nos. 1, 2 & 3								Aulcraft OA38 Polymerin
n–requires catalyst and	Formulated as polymer coating for Styrene								Aulcraft AX8956
s brittleness, checking,	High temp. baking coating for Thermosets 5, 8								Plaskon 3300–3311–3353
., inferior to Melamine-	As base and top coating–force dry–for 1, 2, 3 & 5, 8								Aulcraft AX 8503
–requires catalyst & heat ar resistance over Al.	Formulated as top polymer coat'g–Styrene–								Aulcraft AX 9019

vents (Column 5)

ons	K—Ketones	
ons	W—Water—solution or emulsion	
	V—Varsol (mineral spirits)	

Code for Resistance Rating (Column 6)

●	E—Excellent	P—Poor	✱
●	G—Good		
✱	M—Moderate		
	F—Fair		

the adjoining column.

HOW TO USE THE CHART

is demonstrated by the following example.

A base and top coating of resin lacquer compatible with Butyrate MH flow plastic parts is required for the protection of an evaporated aluminium coating (dip dye or plain).

As a first choice the resin solvent combination, *Cellulose Acetate - Ester - Ketone*, is suggested.

The relevant properties of this material can be traced in the chart by following the symbol ✱. As shown its characteristics are the following:—

1. It is a thermoplastic material.
2. It requires air or force drying for solvent removal.
3. It requires curing up to 70-80°F.
4. Its water and oil resistance is poor.
5. Its solvent (mild) resistance and resistance to handling is moderate.
6. It forms a moderately hard dry film.
7. It shows crazing and penetration of the solvent to the undercoat.
8. It forms a compatible air dry spray top coat.

For the reason of characteristic No. 7 *this material has to be rejected.*

As a second choice a resin solvent combination, *Melamine-Alkyd-Alcohol-Arom. Hydr. Carb.*, is suggested.

The table gives the following characteristics of this material (see symbol ●) :—

1. It is a thermosetting material.
2. It is a polymerising material.
3. It requires curing at a temperature of 130-160°F.
4. It is excellent for water and oil resistance.
5. It is good for solvent (mild) resistance.
6. It tends to blush at high humidity.
7. If a catalyst is used it is durable and mar resistant.
8. It is suitable for base and top coat.

This material is suitable for the purpose if anti-blush addition is specified in its formulation and if a suitable catalyst is provided (see characteristics 6 and 7 respectively).